



THE
ASSAYER'S GUIDE;
OR,
PRACTICAL DIRECTIONS
TO ASSAYERS,
MINERS AND SMELTERS,
FOR THE
TESTS AND ASSAYS, BY HEAT AND BY WET PROCESSES,
OF THE
ORES OF ALL THE PRINCIPAL METALS,
OF
GOLD AND SILVER COINS AND ALLOYS,
AND OF COAL, &c.

BY
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A NEW, REVISED AND ENLARGED EDITION.

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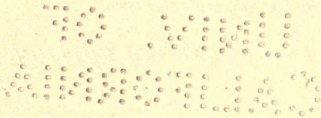
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PREFACE TO THE REVISED EDITION.

THE "Assayer's Guide" has so steadily maintained the popularity and reputation it has enjoyed for many years, and has met with such success, that it has been determined to issue a new edition containing, in addition, numerous recent and approved assaying methods.

The material for this purpose has been largely drawn from the articles on Metallurgy contributed by Prof. Bruno Kerl and others to the fourth edition of "Muspratt's Theoretische, Praktische and Analytische Chemie," now being published in Germany.

While many improvements in the apparatus used in assaying have been made, only a few of the most notable of these novelties have been introduced, it being believed that the implements and utensils described and illustrated in the work are sufficient for all practical purposes.

The book has also been provided with a copious

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iv PREFACE TO THE REVISED EDITION.

Table of Contents and a very full Index, which will render any subject easy and prompt of reference.

In its enlarged and revised shape, it is hoped that the work will fully maintain its claim to the favor it has so long enjoyed as a complete guide in the art of assaying.

PHILADELPHIA, *December 1, 1892.*

PREFACE TO THE ORIGINAL EDITION.

IN presenting this little volume to the public, I trust that I am filling a void in our chemical literature. I believe there is no work in the English language on assaying which combines with practical usefulness a sufficiently popular character for those readers who have not made metallurgy and its kindred sciences objects of their especial study. My desire is to offer a book which fully treats of all the subjects of assaying, and whose price will yet place it within the reach of all persons professionally interested in this important branch of knowledge.

The more compendious works on chemistry, if they allude to assaying at all, give so sparing and incidental remarks, that they do not throw any light upon it for technical purposes, as indeed it does not fall within their province to do.

The immense mineral wealth of the United States, to which the discoveries in California have so largely added, makes this art a very desirable acquirement for every one engaged in any business connected with the metals; and it would appear indispensable to those desirous of deriving the greatest advantage from a residence in that promising and alluring part of our country.

Although these pages principally treat of the processes by heat, I have thought it proper to de-

scribe also some wet processes, either where they serve as methods to rectify the former, or where no others exist; as, for instance, is the case with platinum. For the coins I have selected those employed at mints, and which are therefore generally considered the most serviceable for the analysis of those alloys.

I feel induced here to recommend at least a partial study of blowpipe assaying to those who may wish to make use of the instructions given in this treatise. This infant branch of chemistry was first created, I may say, in Sweden by Cronstedt, and established more firmly by Gahn and Berzelius, both his countrymen, and has of late been made by Plattner a method even for quantitatively ascertaining the contents of most minerals.

The requisite blowpipe utensils for the mere detection of the principal components of ores are so few, can so easily be commanded, and may be so readily carried about one's person, that, at least for the purpose of a prior test, it is very advisable to possess some knowledge of their application, which can be acquired with great facility.

I cannot conclude these brief remarks without seizing upon the opportunity of acknowledging the liberality of a distinguished officer at Freiberg in Saxony, Mr. Fritzsche, chief assayer of that mining district. I have made frequent use of his notes and hints, given me while I had the advantage of a personal intercourse with him.

O. M. L.

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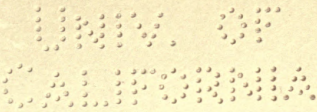
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THE ASSAYER'S GUIDE.

INTRODUCTION

ASSAYING is the science which treats of the various methods of ascertaining the amount of one or of several ingredients of a chemical compound, such as an ore or alloy, but is solely intended for practical purposes. For this reason one of its main objects is to be able to attain to a very great accuracy with the smallest and cheapest means, and in the shortest possible time. In this it is contradistinguished from inorganic analytical chemistry, properly so called. The aim of the latter is to ascertain all the different composing elements of an inorganic compound, and also the exact amount of each,

and for this reason the chemist should never be deterred by a great waste of time and money, if an accurate result should require extravagant means. As assaying is only an aid to technical operations, and these never can extract the whole amount of a metal or other component part contained in a composition, (be the latter a natural or artificial one,) with true chemical precision, it is self evident that much time and labour would be unnecessarily lost, if the same attention were devoted to it as would be requisite in an analysis. This, however, also shows that it is utterly inadequate for purely scientific ends.

Assaying was the earliest known branch of chemistry, and in fact that one which afterward, through the medium of the various investigations of alchemy, drew attention to theoretical chemistry, and thus founded that science.

The art of assaying is so ancient, and it has so constantly and imperceptibly re-

ceived new additions and perfections, that its origin is entirely unknown. Agricola of Saxony, who lived in the earliest half of the sixteenth century, was the first to collect the facts and write on this subject. (*G. Agricola de Re Metallica*, libr. XII. Basil, 1546.) Since then the manifold discoveries of more enlightened periods have vastly enlarged and developed this branch of study.

It is not by any means always necessary that the same measures be employed for these tests as are used in the separation of the single constituent from the rest of the compound in large quantities; as, for instance, in the processes of smelting and amalgamation, although, if convenient, it is very advisable, as it facilitates the metallurgist in the detection of the best fluxes and admixtures to be added to his peculiar ores.

The assayer should be guided in his operations by chemistry, and the study of

the latter, particularly as far as regards the principal and more common substances, should never be omitted. In other respects, all that can be recommended to one who desires to perfect himself in assaying, is the most scrupulous cleanliness, order, and precision as regards his assays and implements, and the most unwearying adroitness and attention in performing the manipulations required by his science.

I.

*Description of the Implements and Utensils
used in the course of Assays by Heat*

AMONG the articles necessary for an assayer's office, of course the first that requires a description is the furnace. For most purposes the *muffle* furnace suffices, and it is the only one used for gold, silver, and copper ores; but for iron, lead, &c. we require another, though much simpler one. I shall first describe the *muffle* furnace, (Plate I. figs. 1, 2, and 3.) The characteristic part, the one from which this furnace derives its name, is the muffle, illustrated on the same plate, in figs. 7 and 8. Both these diagrams give a perspective view, the first a semilateral, the second one from in front. They can be procured in most of our larger cities, and are manufactured in

great quantity in Hussia, of a very firm and fire-proof siliceous clay, the same material as that used for the well-known Hessian crucibles, (Plate II. fig 5.) The one from which the drawing was taken in Plate I. measures eight inches across the bottom from mouth to back outside, and four inches down the back. The holes opening upwards towards the interior measure each an inch and a half lengthways, but their number of course varies according to the size of the muffle, which again should depend on the quantity of assays expected to be performed at once. The back and the mouth of the muffle are equal in size.

The muffle furnace is exhibited from in front in fig. 1, in a lateral section in fig. 3, and in a transverse perpendicular section in fig. 2. A scale, showing the proportionate dimensions in English feet, is attached, to facilitate the use of the drawings in building a furnace; for though very small, they have been made with much care, and, with

the little sketches attached, are fully capable of being employed to that effect. As will be seen from the first figure, the line A B is seven feet long, while the external width of the furnace, C D, is two feet nine inches. The furnace consists of three chief parts, the chimney, *r*, in figs. 2 and 3, (which need only be $3\frac{1}{2}$ feet high if it enters the chief chimney of the laboratory, which should then measure at least 9—12 feet,) the part above C D, in fig. 1, containing the muffle, and the part below that, the grate, ashes box, &c., &c. In fig. 1 we see immediately below the chimney a representation of bricks, (*firebricks*, for all in immediate contact with the heat should be of this kind; the external ones are common burnt bricks.) These, though fastened in tight, are placed so that they can be taken out, to put in a new muffle, or make other repairs when necessary. In this brickwork we have two openings, *d* and *a*. The former is only a small aperture, for insert-

ing pieces of sheet iron, upon which to dry the cupels. It is, however, not immediately necessary, and, if present, is always closed with a tight brick when the muffle is in use. *a* is the opening of the muffle, which can be closed by a piece of fire-proof brick represented in fig. 4, having an iron piece protruding on the exterior, with a narrow opening, into which fits the handle fig. 5, which we apply on removing it. To the right and a little below the muffle is a loose brick, *e*, to be taken out whenever it becomes necessary to remove the fire-proof clay bar, *o*, which holds the muffle, (vid. fig. 2.) As seen in fig. 3, this bar as well as the three others shown in the little sketch affixed to fig. 2, and of which the centre one is a little the longest, are slightly inclined, thus elevating the back of the muffle an inch above the front, and giving a better circulation of heat, as should be the case where stone-coal or cokes are used. In burning charcoal it may be horizontal,

although this inclination has another advantage, inasmuch as it enables us to clean out the muffle easier, should a cupel upset, the contents running forward. The three fireclay bars, *sss*, are loose, while *o* is inlaid on one side, and on the other fastened in with a loose piece of brick and an iron wedge, which can be removed, as already remarked, through *e*. On inserting the bars and muffle, the parts where they meet, and where the former touch the other bricks, should be powdered over with bone-ashes, to prevent a possible adhesion from the intense heat. Small cracks in the muffle should be mended with coarsely powdered burnt clay. *p*, in fig. 2, is the open space left round the muffle about an inch and a half wide, and into which the opening *d* in figs. 1 and 3 leads. Immediately below *CD* we have the door *b* exhibited in all three figures. It is for the introduction of fuel, and is just above the grate, *xx*, in figs. 2 and 3, which consists of six iron bars placed in the same

inclination as the muffle. The door *f*, fig. 1, is of iron, with an inside coating of fire-clay, an inch and a quarter thick, kept in place by iron rivets, thus protecting the assayer from the radiating heat of the iron, which would otherwise soon be white-hot. Below the grate stretches the chamber *q*, in figs. 2 and 3, both for draught and for the cinders. The plane at the bottom of this is inclined like the grate and muffle, and has the opening *c* to take out the ashes. To introduce a regular and sufficient quantity of atmospheric air, the channel *k* has to be constructed, which enters *q* at *h*, and passing under the floor of the laboratory opens outside at *i*, fig. 3: *l* is a small piece of sheet iron, by pulling out or pushing in which, we open or close the passage *k* at pleasure, and accordingly as we require a great influx of air or not, for a high or low temperature. The little figure annexed to fig. 1, shows the construction of the little opening and slide in the lower door. It is

exactly as with a common iron stove, γ being the hole, while the slide $\alpha \beta$ can be closed over it by means of the handle, or removed, as may be desirable for different degrees of ventilation. This furnace is of the kind proposed by Professor Plattner for the Freiberg assay office for the use of stone-coal and cokes, and where it has been employed to great advantage, as regards economy and practicability in general.

Having thus pretty minutely given a description of the muffle furnace, as it can best be constructed when not required to be movable, I shall proceed to the furnace for tests of iron ores, lead ores, &c., also in a shape not intended to be moved, as represented by a lateral section in fig. 6, Plate I. Those parts in immediate contact with the heat should be constructed of fire-proof bricks, which ought to reach some distance up the chimney. The main body of the furnace B and A is either square or round, and twelve inches in diameter. The back

is about three feet nine inches high, while the front measures slightly less, as the top has a little slant, merely for convenience in easier being able to get at the bottom. A is the part where the ashes collect, with a door at H having one or several openings, as is represented in the door *g*, in fig. 1, for the sake of regulating the ventilation. If this furnace is to be used for iron assays, in which case a very intense heat is requisite, a similar channel as illustrated in K, fig. 3, should be attached, the longer the better, and, if possible, opening into some cool place, e. g. a cellar, thus creating a stronger current of air, and consequently a better ventilation. Above A are the bars forming the grate FG, which ought to vary in distance from one another according as we use charcoal or stone-coal and cokes, in the former case being closer to one another. B is the part in which the crucibles are placed, as well as the fuel, and which has one opening for the introduction of the same

with the cover E, suspended, when open, by a chain, but which is shut when the furnace is in use. This cover is commonly of iron, covered with fireproof clay.

C is the channel leading off to the chimney D, which may vary in width from one half to one quarter the width of B. For assays of lead and copper it is not necessary to have the chimney very high, as no very severe draught is required; but for iron assays it should be about thirty feet. In the assay office in Clausthal it measures forty-eight feet. In this case, of course, if there is also a muffle furnace on the same hearth, it is best to conduct the chimneys of both into one large one. On placing the crucibles in this furnace—twelve have room in it at once—a piece of firebrick, K in the drawing, is first placed on the grate, which, to avoid adhesion of the crucibles, may be powdered with bone-ashes. This brick is employed to have the crucibles more in the focus of heat, by elevating

them above the coal around it, and also—clay being a bad conductor—to keep them from immediate contact with the cold blast. The fuel should never be heaped above 1, the mouth of the channel C, and large coals should be placed below, to keep the grate from choking. If several crucibles are used at once, they ought to stand sufficiently apart to admit coals between them, as they otherwise might melt partially in the course of the process, and form one compact mass.

On the foregoing pages I have furnished descriptions and measurements of furnaces, which, as I have already remarked, though easily constructed, are not portable; and it is proper that, before closing this chapter, I should devote a few lines to the mention of those, which, from their being movable, and thus obviating the difficulty and inconvenience of constructing one, might, in many cases, be preferred, although their smaller dimensions, as a matter of course, make it more difficult to perform good

assays, and impossible to attend to many at a time. Among these we have both muffle furnaces and wind furnaces such as last described. Many forms have been manufactured, and it is not necessary to enter into detailed descriptions. They can generally be procured at all larger stores of chemical apparatus. The *Battersea* furnace is of fireclay made in sections and bound with iron bands. *Brocon's portable assay furnace* consists of a sheet-iron frame 27 inches high and 14 inches square, lined with firebrick, the interior being smooth and straight from top to bottom. The cover is ridged to lessen the danger from cracking. The muffle door is cast iron and is fitted with a circular opening filled with mica, that the operations going on within the muffle may be seen when the door is closed. After having thus given a description of the furnaces used in the course of the experiments elucidated in the following pages, the reader's attention is called to other instruments used

in the assays of ores. Among these, probably the balance will appear most conspicuous. There ought to be two in use, one very accurate, the other less so, for more common purposes, as weighing off the ingredients or admixtures, such as pure lead, borax, &c. The other one should be made with great care, and only used to weigh off the small buttons of gold and silver procured. Before weighing the balance must be tested as to its equilibrium. The substance to be weighed should be cold, and must not be placed directly upon the scale pan, but upon suitable smaller pans, watch glasses, etc.; hygroscopic substances in closed tubes. The balance beam should be raised from the knife every time before a weight is put into or removed from the pan. The weights must not be put in the pan at random, but systematically, this being the only way of saving time. The highest probable weight should be added first, then the next lowest, and so on until the equilibrium has been established.

The pans should then be changed in order to test the correctness of the weight. Perfect equilibrium of the balance is, however, not absolutely essential, as a correct weighing may be obtained by placing the substance to be weighed in one pan and in the other pan any convenient material as a make-weight, such as tin-foil, shot, granules of lead, etc., until equilibrium is established. The balance is then raised, the weighed substance is removed from the pan, and sufficient weights to counterpoise the balance are put in its place. The sum of these will give the correct weight of the sample. It is a good plan to *always* place the weights in one pan (the right-hand one) and whatever is to be weighed *always* in the other.

In the choice of a scale for weighing larger quantities (fluxes, etc.) much latitude may be allowed. It need not be very delicate; a grocer's scale will do.

We must now cast a rapid glance at the

smaller and less costly utensils, but all of which are necessary for the accomplishment of the operations to be performed as directed in the following pages. I allude to the crucibles, *cupels* or cups, and tongs, &c., illustrated in Plate II. The former, figs. 1, 2, 3, 4 and 5, are all of their natural size in the drawings. Figs. 1, 3, and 4, should be made from accurate descriptions by a potter, of a pure clay, containing as little silica mechanically combined as possible, since its presence prevents the vessels from being sufficiently porous to allow the oxidizing metals to enter, which would, as will hereafter be seen, become a great drawback to the assay. Some hundreds must always be kept on hand, for they can only be used once, as the lead, almost always present, glazes them immediately.

Fig. 1 represents a *cupel* used in the operation technically termed *cupellation* of the gold and silver ores: *a* shows it from above, *b* from the side. The concave inner

surface should not be too shallow, thus letting the contents run over; nor, on the other hand, too deep, in which case too small a surface would be exposed to the oxidizing power of the heat. The line given in *b* shows the proper inclination within.

Fig. 2 is a truncated cone, with a very slightly concave surface at the wider extremity or top. It is made in great part of bone-ashes, being among the most porous substances that can stand a high temperature. I give here a recipe for their preparation, furnished me by Mr. Fritzsche. It shows the relative quantity of the ingredients as used for these cupels at his office, where they are made by the laboratory servant in wooden moulds, using a rod, shaped like a pestle, to make the hollow at the top. The mass consists of 4 parts of wood-ashes previously freed of the pot-ash by filtration, 1 part of lime, and 1 part of bone-ash.

Fig. 3 shows a flat, shallow crucible made of the same pure clay as that represented in fig. 1: *a* is the cup as seen from above, while *b* gives a lateral view, showing also the curve of the inner surface. This vessel is used in the assay of copper, as well as fig. 4. Fig. 3 is termed in German "*Kupfer Garscherbe*," from the fact that with its use the copper assay is finished.

Fig. 4 is a high crucible, as mentioned above, used for copper and lead. It is made of the same material as the former. The reason of its having so great a height compared with its width, and otherwise so peculiar a form, is to concentrate all the heat round about the bottom of the interior. In some places, in lieu of better, the Hessian crucible (fig. 5) is used; but No. 4 is much better adapted, from the height of the foot-piece, which also protects the contents from the cooling influence of the iron bars on which they are placed,

and from a too direct contact with the cold draught. Some assayers use an extra cover for this crucible, a very superfluous addition, since the bottoms of the old crucibles answer admirably as covers to the new. As mentioned before, these also are useless after once having been employed, and before throwing them away it is therefore well first to break off the feet.

Fig. 5 gives a drawing of a Hessian crucible, which is used chiefly in the assays of iron ores. These are imported into the United States, and can be procured in almost every crockery store. Their dimensions are very different, and so contrived that one fits into the next in size, being in sets of about a dozen. They are commonly triangular above, and round below, as shown in the figure; but sometimes they are made round above and below, and where these can be had, they should be preferred. If they cannot be procured, similar crucibles of pure clay may be sub-

stituted, and in fact they are sometimes considered preferable. The latter are then commonly made with a foot-piece, somewhat as in fig. 4, though not quite so strongly separated from the main bulk of the vessel. These or the Hessian crucibles, as will be seen hereafter, should be kept constantly on hand thickly coated with charcoal. They are prepared in this manner. Charcoal of any oak-wood, except red-oak, which contains much oxide of iron, is the best on account of its density. It should be pulverized with the utmost care, since a coarse powder will never furnish a consistent mass. To this we carefully add water until we procure a tough paste or dough, and with this fill the crucible very tightly, always ramming down every new portion introduced, with a pestle or piece of wood. When the whole interior is filled up, we cut out a hole in the middle with a penknife, leaving a margin of about half

an inch at least, below and all round. (See Assay of Iron Ores.)

Fig. 6 furnishes a sketch of an implement, which, though not immediately necessary, will be found extremely useful, inasmuch as it tends to keep up order and accuracy, great requisites for an able assayer. As will be seen from the different views *a* and *b*, it consists simply of a piece of sheet-iron, having a wooden handle and several semi-circular cavities, the number of which may vary according to the number of assays expected to be performed at once. If a large muffle is used in the furnace, it is well also to have this large; and, in fact, I would suggest having the number of holes in each row equal to the number of cupels, of the sort illustrated in fig. 1, that can, without collision, be placed across the muffle from side to side. (See Silver and Gold Ores.) This pan, or whatever we may otherwise term it, is used to cool the buttons of metal and

their surrounding slag after having oxidized part of the lead, &c., and separated the gangue-rock, as is the case with silver and gold (q. v.) The holes should be $1\frac{1}{2}$ inch wide and $\frac{1}{2}$ inch deep:

Figs. 7 and 8 represent two different kinds of tongs. Fig. 7 is only shown in a lateral view, since its characteristics are thus exhibited, while fig. 8 is sketched from above. They are both very necessary in the progress of our investigations. The first is shaped like common fire-tongs, only that the ends, instead of widening into a flat circular lobe, are bent downward like a poker, thus enabling us to handle the crucibles with great care. The second tongs has each piece ending in a semi-circle, the diameter of which circle, when united, should be so that it may exactly embrace the lower part of fig. 1. It is used to place those cupels into the muffle, as we are thereby prevented from touching the contents. In using this latter in-

strument great caution should be taken not to upset the cupels. Let me first remark, that on introducing the cupels into the muffle, we always commence with No. 1, i. e. first assay on hand, behind on the left. It may seem a very superfluous precaution to dwell thus long on so simple a manipulation as the one I am now describing; but I know from experience, how many failures carelessness in this procedure occasions to the uninitiated. Let us now suppose, that we have the crucible, fig. 1, filled with the powdered mineral to be tested, and the other additions, such as lead or borax. Grasping the cupel firmly at the bottom, we lift it up high, so that, when pushed into the mouth of the muffle, it is a good deal above its floor, and cannot knock against it. As soon as it is within the muffle, without leaving hold of it, and keeping it up high, we hold the side of the tongs up against the side of the muffle to steady them, then run them along quietly

though not too slowly, for that only causes the hand to tremble, until the crucible is just above the spot where we intend to place it, (when once down it should never be moved,) and then slowly lower it, not letting go till it stands secure.

These tongs should be made 'light enough conveniently to handle them with one hand. The handles are made of some good firm wood, though No. 8 is generally held lower down than the handles, and, I may add, never so that the end of the tongs, the handle, passes under the arm, but exactly as the savages hold their darts when about to hurl them. It may seem awkward at first, and yet I know many accidents, as regards the assays, having occurred from not attending to it. The length of the tongs ought to be about three feet. It may be well to supply oneself with a third pair, with straight and rather pointed ends.

For sifting brass or copper wire sieves are generally used in preference to hair sieves, since the latter deteriorate. Such sieves are furnished of various diameters (6 to 8 inches is a good size). For less valuable ores the sieves have about 35 to 50 meshes to the square inch and for more valuable ores from 70 to 80. The box-sieve of tinned iron consisting of a sieve, of about 80 to 100 meshes to the linear inch, with tightly fitting bottom to catch all the sifted material, and cover to retain the dust, is a valuable implement. Brittle substances will pass through the sieves without difficulty, but those with malleable admixtures will leave behind a flattened residue, as, for example, ores carrying native silver and copper, silver glance, granules of lead in slag and thin matt, sweepings containing gold and silver, etc. In case hard gangue (quartz) is to be sifted, the fine mass which has passed through the sieve is several times rubbed together with the coarse residue re-

maining upon the sieve, until everything has passed through. The residue of soft gangue is weighed and at once assayed by itself, and the fine siftings separately also, after they have been mixed upon glazed

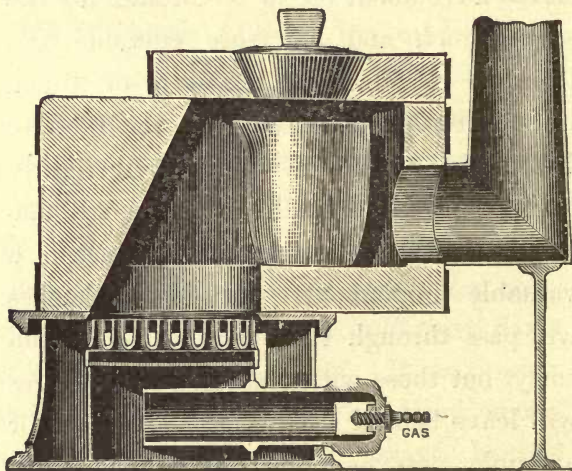


FIG. A.

paper and several times passed through a coarse sieve. The entire yield is then calculated by adding the product of both together.

Gas assay-furnaces. These furnaces have

been known and used for some time. The source of heat is our common illuminating gas. Fig. A shows Fletcher's direct-draft crucible furnace. It consists of a fire-clay body held together by sheet-iron bands. The heat and flame pass through the body of the furnace to the chimney. It can be used either for scorifying or cupelling, and by removing the top cover the heat has full play upon a roasting dish placed upon it.

Fig. B gives a representation of a group of gas furnaces designed by Walter Lee Brown and described by him as a complete gas-assaying plant.*

Its duty is to do roasting, crucible fusion, scorification and cupellation. The furnace at the left is for roasting sulphurets or other ores for experimentation or actual work. It is what is known as a Fletcher No. 163, the same as shown in fig. A. The heat and flame pass from the burner through the

* Transactions of the American Institute of Mining Engineers, 1884.

furnace, and out and up the chimney pipe.

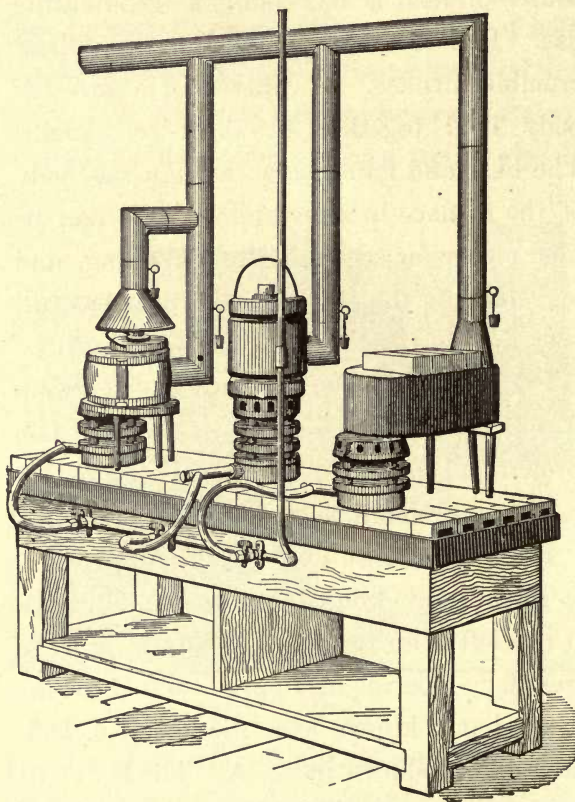


FIG. B.

The funnel-shaped pipe over the cover is to

catch and draw the fumes up the chimney. When the burner is lighted a powerful draught ensues, carrying all odors and fumes at once away. Both the pipe and hood are provided with dampers, controlled by small weights. The burner is connected to the gas-tap by stout $\frac{1}{2}$ inch rubber tubing. A cast-iron tripod supports one end of the furnace and keeps everything firm.

Next in regular order (supposing a sulphuret ore to be under treatment) is the middle furnace for crucible fusions. This, likewise, consists of a furnace and burner. The latter is a Fletcher No. 15, of the same construction, however, as the No. 16. The furnace proper is made in five parts, the central section (a cylinder of fireclay), the bed-plate upon which it rests and which has an opening for the flame to pass through; the cover (with handle attached), which also has an opening filled by a plug—all of fireclay; and finally a plumbago lining. The

rubber tube chimney connections and damper are like those of the other furnace.

Finally at the right is shown the furnace for scorification and cupellation, which the author designates the "Monitor." Fig. C

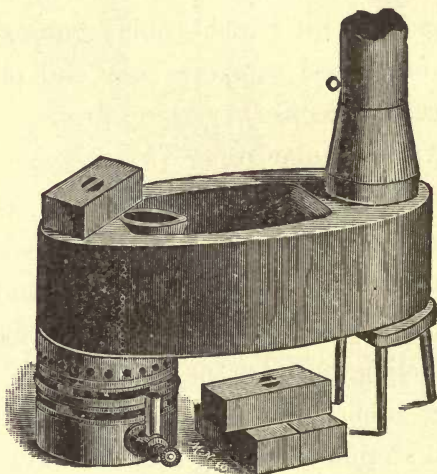


FIG. C.

shows it enlarged and uncovered. Its form is almost that of a reverberatory furnace, the movable bricks, when in place, being the roof. Looking at it from another point of view, it may be considered as a muffle

with the flame as well as the heat inside. Its exterior dimensions are as follows: length 20 inches; width 7 inches; and depth $5\frac{1}{2}$ inches. In the interior, upon the bottom, are four little wedge-shaped bridges of fire-clay which are movable; and upon these rests a false bottom or floor, also movable. The latter corresponds to the muffle-bottom of an ordinary furnace, and upon it is done all the work. It is $3\frac{1}{2}$ inches wide by $7\frac{1}{2}$ inches long and $\frac{1}{2}$ inch thick, and has a shoulder or bench running across its entire width on the end nearest the burner. The covering bricks, four in number, are each 7 inches long by $2\frac{3}{4}$ inches wide and $1\frac{3}{4}$ inches high, each with a slotted bridge for convenient handling. The connections are similar to those of the first mentioned furnaces.

The 3-inch stove-pipes of all three furnaces are fitted into one long, horizontal pipe, which fits snugly into the chimney. The bench or table upon which rest the

furnaces described is made of pine, well seasoned and firmly joined, to resist as much as possible the warping influence of heat, and to support the weight of the furnaces and table-tiles. In dimensions (not figuring on the top, which overlaps 1 inch all round) it is 4 feet 6 inches long, 1 foot 7 inches wide, and 2 feet 1 inch high, plus the thickness of the top, which is $1\frac{3}{4}$ inches. A double coat of shellac varnish is its sole ornamentation. To the sides and ends of the table-top are firmly screwed four strips of band iron of $2\frac{1}{2}$ inches width and $\frac{1}{8}$ inch thickness, and of such lengths as to alternately overlap at the angles, making smooth joints. The top of this sort of wall is $\frac{3}{4}$ inch above the bed of the table. Upon the latter are 114 fire-clay tiles, or, rather, clamps, such as are used to join house tiles and having the shape of the letter "E" less the middle projection. Their average size is $3\frac{1}{2}$ inches by 3 inches across and $1\frac{1}{4}$ inches high. They are so arranged on the

table as to leave a series of six air tubes or chambers running its entire length. The spaces between the tiles are filled with a mixture of plaster of Paris and Venetian red rubbed up with water. The latter color is also used for the tiles themselves, and somewhat on the fire-clay portions of the furnaces.

A $\frac{3}{8}$ -inch bore gas-pipe, with proper taps and nozzles, is screwed to the front of the table.

The manner of operating the furnaces is simple. As regards the roasting-furnace, it is necessary merely to shut off the dampers of the other furnaces, turn on and light the gas and regulate the heat to suit the particular ore. The control of the mixture of gas and air is effected by means of the milled handle at the burner. In using the crucible-furnace, remove cover, turn gas on full at tap, light and regulate by milled handle. Crucibles containing charges that are to be heated gradually can be placed in

the furnace as soon as lighted ; others after the lapse of a few moments to allow the furnace to become thoroughly heated. Placing the charges in cold, good fusions of refractory ores have been made in twenty-five minutes from time of lighting. The furnace will take crucibles in size up to Battersea "S" ($4\frac{1}{2}$ inches across by 5 inches deep).

To manage the "Monitor," remove the covering bricks, open the damper and shut those of the other furnaces, turn back the milled handle at the gas-entrance to the burner so as to allow a full flow of gas, turn gas on full at tap, light and put back the bricks into place. In from twelve to twenty minutes, the interior will be hot enough for work. The bricks are then again removed, the charged scorifiers are placed on the false floor, the bricks are replaced, and the excess of gas is turned off at the burner. When the charges have melted, slide the bricks aside more or less

—principally those nearest the burner—to admit air for oxidation. In cupellation, the gas is turned down more than in scorification.

The time of performing either scorification or cupellation varies according to the nature of the ore, charge, size of button, etc., but is about the same as that occupied in the use of a coke furnace. The consumption of gas is not far from thirty cubic feet per hour. It is not intended or claimed that this furnace can take the place of one required to be run from ten to twelve hours per day; for that, of course, solid fuel will be cheaper. But for short runs of from one to say four hours, it is economical, as are also the others here described. The complete plant, as illustrated, costs about \$75.00. The furnaces are manufactured by the Buffalo Dental Manufacturing Company, of Buffalo, New York.

II.

Re-agents Used in Assaying.

Re-agents for dry assays. According to their action they may be divided into:—

1. *Reducing agents.* *Wood charcoal*, in the form of powder, or of small pieces. One part of ordinary charcoal will reduce from 22 to nearly 32 parts of metallic lead from litharge, according to the purity of the charcoal. When employed in the roasting of antimonial and arsenical gold and silver ores, charcoal exercises a desulphurizing action.

Powdered coke, anthracite and graphite may also be used instead of powdered charcoal, but they are less combustible. *Argol* (*crude bitartrate of potash*) yields considerable carbon on becoming carbonized, and in consequence exerts a vigorous reducing

effect, but causes refractoriness. For this reason its content of carbon is reduced, if necessary, by mixing it in different proportions with saltpetre. The mixture is poured into a red-hot crucible, placed under a well-drawing chimney. The mixture deflagrates and emits empyreumatic odors, and a partial oxidation of the carbon taking place, a mixture of potassium carbonate and carbon is formed. This is known as *black flux*. For vigorous reduction it is made from 3 parts argol and 1 saltpetre; for less vigorous, either $2\frac{1}{2}$ and 1, or 2 and 1 parts respectively. *White flux* consists of 1 to 2 parts saltpetre and 1 part argol; *gray flux* of 3 parts argol and 2 saltpetre. On account of the hygroscopic properties of black flux, a mixture of potassium carbonate or of bicarbonate of soda and flour is used in preference, it being also cheaper. Usually 20 to 25 per cent. of wheat flour is taken, but for more vigorous reductions, 30 to 35 per

cent., and even as much as 50 per cent.

Potassium cyanide is an energetic reducing (as well as desulphurizing) agent. It being very poisonous, great care should be exercised in handling it.

Potassium ferrocyanide yields, on heating, a mixture of iron carbide, ferrous and ferric oxides, free carbon, and a small quantity of potassium cyanide. It exercises also a vigorous desulphurizing action.

The following list of the reducing powers of various reducing agents will be found very useful. The values are approximate only, but for all practical assaying they are sufficiently near.

| 1 part of | will reduce parts of metallic lead. |
|-------------------------|--|
| Ordinary wood charcoal, | 22 to 30 |
| Powdered coke, | 24 |
| Powdered hard coal, | 25 |
| Powdered soft coal, | 22 |
| Wheat flour, | 15 |
| Cornstarch, | 11½ to 13 |
| Laundry starch, | 11½ to 13 |

| 1 part of | will reduce parts of metallic lead. |
|-------------------------|--|
| Pulverized white sugar, | 14½ |
| Pulverized gum arabic, | 11 |
| Crude argol, | 5½ to 8½ |
| Cream of tartar, | 4½ to 6½ |

2. *Oxidizing agents.* *Saltpetre.* Ordinary saltpetre of commerce is used. It should, however, be as free as possible from sulphates. Pulverize finely, and keep dry.

Litharge (*plumbic monoxide, yellow oxide of lead*) exerts an oxidizing effect upon metals and metallic sulphides, as well as upon organic substances. It should be dry, and free from any considerable amount of red oxide of lead, as this causes oxidation of silver and consequent loss. When the litharge contains silver, *white lead* not adulterated with heavy spar may be substituted for it. Litharge *entirely free* from silver may be prepared by oxidizing the purest Pattison or Villach lead by cupellation, or bringing such lead, after it has been granulated into fused saltpetre; or by

gradually scattering powdered charcoal upon litharge fused in a refractory crucible, whereby some lead will be reduced which, in subsiding, withdraws the silver from the litharge.

3. *Solvent agents.* a. *Acid*; such as *silica* and *glass*. *Silica* is best used in the form of pulverized silica, since it is then in a very fine state of division suitable for intimate mixture with ores and fluxes. It should be perfectly dry.

Borax-glass. This is produced by fusing borax in a clay crucible, and then pouring it upon a bright metallic surface. It is more fusible than glass, and the boric acid forms combinations with nearly all the bases as well as with silicic acid.

Salt of phosphorus (sodium-ammonium-hydrogen phosphate) or microcosmic salt. In the anhydrous state it is a more energetic solvent agent than borax.

Clay, such as kaolin. Most varieties of clay contain over 50 per cent. of silica.

b. Basic. Potassium carbonate. Ordinary carbonate of potash. It should be ground to a fine powder and kept from the air, as otherwise it would rapidly absorb moisture.

Sodium carbonate (bicarbonate of soda) acts somewhat less energetically than potassium carbonate, and consequently a larger quantity of it must be used. It is less deliquescent, more fusible and cheaper. A mixture of 13 parts of dry potassium carbonate and 10 of calcined bicarbonate of soda furnishes a very readily fusible flux.

Caustic alkalies act more energetically than carbonates, but exert a very injurious effect upon the crucibles.

Calcium carbonate, in the form of chalk (or calcite), may be used for smelting operations at higher temperatures (for instance, in assays of iron).

Fluor-spar is more readily fusible than calcium carbonate, and is especially effective for removing silica. It readily fuses

down with calcium phosphate, heavy spar and gypsum.

Lead oxide (litharge, minium, white lead) readily fuses with silica, the alkalies, and most of the heavy metallic oxides, but less so with the earths and alkaline earths.

4. *Desulphurizing or precipitating agents.* *Iron* in the form of iron filings and as pieces of wire.

Potassium cyanide and potassium ferrocyanide (see p. 56).

Caustic alkalies and carbonates decompose metallic sulphides. The metal is separated, and sulphites, hyposulphites, and sulphates of the alkalies, together with alkaline sulphide, are formed. The latter forms a sulphur-salt with one part of the metallic sulphide, which, as a general rule, can be decomposed with iron.

Carbon promotes desulphuration (black flux, potassium carbonate and flour). *Salt-petre* oxidizes metallic sulphides, while the metals are separated.

5. *Sulphurizing agents.* *Sulphur* in the form of flowers of sulphur; or of iron pyrites.

6. *Concentrating fluxes.* *Lead* in a granulated form (assay lead). *Silver* for alloying with gold (quartation). *Gold* for collecting copper (assay of nickel and cobalt). *Copper oxide* for tin. *Iron pyrites* as collecting agent for copper (assay of matt).

7. *Decomposing and volatilizing fluxes.* *Charcoal and graphite* for decomposing sulphates, arseniates and antimoniates by roasting. *Ammonium carbonate* for decomposing sulphates, especially copper sulphate, at red heat, but less completely lead and bismuth sulphates. *Common salt* for the volatilization of antimony and arsenic in refining black copper according to the Cornish method.

8. *Air-excluding fluxes (covering agents).* *Decrepitated common salt*, as free from sulphates as possible, fuses easily and becom-

ing very thinly-fluid washes down particles of metal adhering to the sides of the assay vessels. *Refined slag* from charcoal iron blast furnaces.

Re-agents for wet assays. The following are principally used :

1. *For assays by gravimetric and calorimetric analysis.* *Acids:* Hydrochloric, sulphuric, nitric and acetic; aqua regia. *Bases and salts:* Caustic alkalies, alkaline carbonates, potassium chlorate, ferrous sulphate, sodium sulphide, etc. *Metals for precipitation:* Iron in the form of wire, or in a pulverulent condition, for copper; zinc in the form of wire or granules, or in a pulverulent state, as a reducing agent for iron solutions, etc.; copper; bromine for decomposing sulphurets, compounds of gold, etc.

2. *For volumetric assays.* Potassium permanganate; sodium sulphide; potassium cyanide; barium chloride; potassium iodide, free and with dissolved iodine; sodium

hyposulphite; ferric chloride; sodium chloride; potassium sulphocyanide, etc. *As indicators:* Litmus tincture, Brazil wood tincture, etc., for acids and alkalies; the salts of iron, nickel and lead, and sodium nitroprusside, for sulphur; starch-paste for iodine; potassium sulphocyanide, etc., for iron oxide.

III.

Assay of Silver Ores by Heat—Cupellation.

SILVER occurs in nature chiefly in lead ores, (galena,) and native, as in the Lake Superior copper mines, or less commonly as chloride of silver, as is the case in the extensive mines of Chili. The following directions, however, refer equally to the assays of all.

As with all minerals to be investigated by these methods, the ore must previously be reduced to the finest possible powder, for which purpose, where many assays are made, a large iron bowl is used as a mortar, the pestle having a wooden handle attached at right angles to the iron part, enabling us thus to employ a much greater force than if it were straight, as commonly is the case. The usual shape of a mortar,

used in kitchens is not so good, for its depth prevents its being cleaned sufficiently to insure accuracy. In lieu of the former, and when small quantities only are desired to be pulverized, an agate mortar may suffice. Of the ore we weigh off one part,—about 3 grammes, equal to 46 grs. Troy, (say 50 grs.) is a good standard weight,—and mix it with ten parts by weight of pure lead, and from one-tenth to one-fifth part of borax, in the manner described below.

The lead is added to extract all impurities, such as copper, nickel, &c., and in cases where these occur in great abundance, should be used in the proportions even up to fifteen and twenty parts, though it is always injurious, and tends to create a loss, to add too much.

To the rules for testing the quality of alloys of silver and copper, will be found subjoined a table by Erker, to regulate this.

The lead of commerce contains many im-

purities, in the shape of other metals, which, since silver always is among them, is very injurious to our assays, and it is therefore necessary to obtain a purer article. At silver furnaces lead is produced as an extra product, sufficiently pure for our purposes. That, for instance, thus procured from the smelting works at Freiberg in Saxony, and used there by the assayers, contains, to 6.40000 parts of lead, from 0.00001 to 0.000015 parts of silver, too slight an impurity to affect the result of the assay. If so situated as not to be able to get it in this manner, the best way is to reduce it by a galvanic process from acetate of lead. This, the sugar of lead of commerce, we can readily dissolve in lukewarm water, after which we should filter off the solution, and introduce a rod of zinc, by which process all the lead is reduced and collected round that metal. After some time remove it, and continue in this manner until all the lead is pre-

cipitated. It is then to be repeatedly washed with care to remove the acid, and afterward dried between blotting paper. If not in a sufficiently pulverized shape, it need only be shaken a little in a towel and finally sieved. A spoon containing a given amount—five parts, equal to 250 grs., is convenient—is used to measure the quantity for each assay, thus avoiding the tedium of weighing each time, a matter of considerable consequence in an assaying office, where frequently hundreds of assays are made in a day.

The vessel into which the mineral, together with the lead and borax, is placed, to be introduced into the muffle of the furnace, is represented in fig. 1, Plate II. (See the description given a few pages back.) We first put one-half of the lead to be used in it, and on the top the silver ore, mixing the two with great care, not to spill the least particle, and afterward cover it with the rest of the lead, and

sprinkle the borax over the whole. The latter is used to facilitate the melting of the lead, and to produce a good slag. When much tin, zinc, or lime is present in the ore, the borax should be increased in quantity up to the largest amount before mentioned.

If we desire to test ores containing very little silica, and our crucibles are not as perfect as we might wish them to be, it is very advisable to make use of some quartz or glass-powder, to be added immediately, or, better still, when the slag is forming, and the process of calcining is thus at an end.

We should, for the sake of economy with the fuel, make several assays at once, and always five or six of each ore, afterward taking the average, and thus regulating our own labour.

After previously firing the furnace, and bringing the muffle to a regular and constant red-heat, we may introduce the cru-

cibles. To quicken the melting of the lead, we place some pieces of well-dried charcoal, which should always be kept on hand, in front of the crucibles. Vapours will be observed to be emitted, caused by the discharge of volatile substances. The door of the muffle, previously closed, should be kept perfectly open as soon as the operation of calcining or roasting the ore has properly commenced, to admit fresh air, and prevent the heat from increasing too much.

This process generally lasts about twenty minutes, after which, by introducing fresh coals into the muffle and again closing the door, we raise the heat, and continue it sufficiently long to let the slags collect and flow easily, so as to make a crust round the white-hot metal. When not employing a sufficiently high temperature, the slag will remain thick or tough, and retain the metallic alloy of silver and lead in small particles disseminated throughout the mass.

From the circular portion of argentiferous lead, which appears to be in a constant rotary motion in the centre of the slag, vapours of lead rise incessantly. The time that should be occupied by this process of melting varies much according to the character and quality of the ore, which sometimes, though rarely, may even require something more than half an hour. Generally much less time is wanted with a good fire, and the assayer can always see at what time the encircling slag has been properly separated from the metal, and even with very little practice is enabled to regulate it. We now again open the muffle, and by oxidizing some more lead during the space of ten minutes, cover the metal entirely, and after that, once more apply a strong heat for a few minutes to make the slags flow easy, and then taking out the crucibles, pour the whole contents into the different hollows of the iron plate, illustrated by fig. 6, Plate II. This should be done very

quickly, for otherwise the lead will not collect in one button, but be distributed in small particles all over the molten slag as above. A little practice will soon accustom any one to all these operations.

On cooling, with a stroke or two of the hammer we separate the slags from the metallic buttons, and with a few more easily shape the latter into cubes as regular as possible.

The crucibles shown in fig. 2, Plate II. come into use at this part of the assay. If well made and dried, they should be very porous, and so friable as to be crushed between the fingers with ease.

In these cupels we expose the metal cubes to a moderate red-heat, which we are enabled to regulate by inserting cold pieces of pottery—old crucibles will be found very serviceable—and leaving the furnace open. The rising vapours are from the lead, and continue until that peculiar bright flash of light is observed, termed *silberblick*, (silver-

gleam,) in German mining and smelting technology, and from which we know that the silver is pure. The utmost attention should be paid to this part of the assay, to ascertain the moment when this occurs. The coolers are then carefully removed, the aperture closed, and the most intense heat possible kept up for a few moments. To do this well is, I may say, the *chef d'œuvre* of an assayer, and on it depends, in a great measure, the accuracy of the assay; for the silver, when pure, requires a much greater heat to bring it to the melting point than when it contains even a very small admixture of lead; and for this reason, if we do not instantly elevate the temperature when the ceasing of the rainbow colours and the bright light of the globule of pure silver inform us that all the lead has left,—then the silver would cool suddenly, and, of course, quicker at the surface than in the interior or at the bottom, and by this subitaneous contraction cause a spattering

motion of the yet unconsolidated particles, and be productive of great loss to the assay.

There is an old adage current among German smelters and assayers,—probably known to all who may have had the advantage of studying this art in that country, where it has been brought to such perfection,—which, since it is many centuries old, shows how long it has been regarded as the great aim, in this mode of testing silver ores, to keep up a moderate temperature as long as any lead is left, and to apply intense heat as soon as all has departed. I insert them here :

“Kühl getrieben, heisser Blick
Ist des Probierer's Meisterstück.”

In English it would be: To evaporate coolly and to use heat when pure, is the masterpiece of an assayer.

When the buttons of pure silver have cooled, we grasp them firmly with a pair of pincers, tight enough to compress the

sides, thus exposing the bottom more, and with a wire brush remove the adhering particles of litharge, and dust of the cupel. Turning them half round so as to make the base a square, we repeat the operation, and then weigh, but only when quite cold, since otherwise the heat, by increasing the bulk, might tend to create a variation.

The silver produced in this manner, it should be remarked, is not perfectly pure, chemically speaking, but the slight impurities are too insignificant to deserve any attention from an assayer, who has nothing but technical purposes in view, though they might require it from a scientific chemist; and for this latter reason, I have thought proper in the fourth chapter to suggest a method to ascertain the exact amount of silver in this button, should it be deemed necessary, though, I must add, that such precision can only be required at mints, if the investigations are not con-

ducted for science' sake, in which case, the wet process would, of course, be preferred from the beginning.

Besides this, small inaccuracies in the shape of losses, as has already been remarked, cannot be averted, even though the assay be performed with the greatest care, as the evaporating, or rather oxidizing lead probably carries off small particles of silver, particularly when too much heat is used during that process. It is impossible to give any perfect rules how to obviate such faults, since so much depends on the care and attention paid, as well as on the acquired practice and innate practical skill of the assayer; yet, pre-supposing all this as perfect, the Parisian mint has established a table to regulate their assays, which will be found on pages 77 and 78. From this it is seen that the different losses with different quantities of silver vary considerably, nor is the loss a per-centage, being greatest where the original or true

amount of silver in the ore or alloy is seven hundred.

This table should always be referred to, as it enables us to calculate the true amount of silver, and to rectify those unavoidable faults which may be occasioned by the lead carrying off portions of the silver entirely, or else drawing them along with it into the pores of the crucible.

IV.

*A Table to rectify the Loss incurred during
the Silver Assay.*

| Real amount of silver. | Amount of silver found by the assay. | Loss during the process. |
|---------------------------|---|-----------------------------|
| 1000 | 998·97 | 1·03 |
| 975 | 973·24 | 1·76 |
| 950 | 947·50 | 2·50 |
| 925 | 921·75 | 3·25 |
| 900 | 896·00 | 4·00 |
| 875 | 870·93 | 4·07 |
| 850 | 845·85 | 4·15 |
| 825 | 820·78 | 4·22 |
| 800 | 795·70 | 4·30 |
| 775 | 770·59 | 4·41 |
| 750 | 745·48 | 4·52 |
| 725 | 720·36 | 4·64 |
| 700 | 695·25 | 4·75 |
| 675 | 670·27 | 4·73 |
| 650 | 645·29 | 4·71 |
| 625 | 620·30 | 4·70 |
| 600 | 595·32 | 4·68 |
| 575 | 570·32 | 4·68 |

| Real amount of silver. | Amount of silver found by the assay. | Loss during the process. |
|------------------------|--------------------------------------|--------------------------|
| 550 | 545·32 | 4·68 |
| 525 | 520·32 | 4·68 |
| 500 | 495·32 | 4·68 |
| 475 | 470·50 | 4·50 |
| 450 | 445·69 | 4·31 |
| 425 | 420·87 | 4·13 |
| 400 | 396·05 | 3·95 |
| 375 | 371·39 | 3·61 |
| 350 | 346·73 | 3·27 |
| 325 | 322·06 | 2·94 |
| 300 | 297·40 | 2·60 |
| 275 | 272·42 | 2·58 |
| 250 | 247·44 | 2·56 |
| 225 | 222·45 | 2·55 |
| 200 | 197·47 | 2·53 |
| 175 | 172·88 | 2·12 |
| 150 | 148·30 | 1·70 |
| 125 | 123·71 | 1·29 |
| 100 | 99·12 | 0·88 |
| 75 | 74·34 | 0·66 |
| 50 | 49·56 | 0·44 |
| 25 | 24·78 | 0·22 |

In Freiberg somewhat different results have been obtained. With refined silver the loss by absorption by the cupel was found to be 0.0015 to 0.0002; and in alloys of medium richness the loss was greater than that stated in the preceding table. For instance, with 750 thousandths and 16 weights of lead, the loss was 5.55 thousandths, but with 11 weights of lead it accorded with the table, 4.52 thousandths. According to Plattner, fine silver with five times the quantity of lead frequently gives a loss up to 0.009, refined silver with 937 thousandths and five times the quantity of lead, 0.0042 to 0.0059; refined silver with 687 to 750 thousandths and 14 times the quantity of lead, 0.0073 to 0.0083.

V.

Gay-Lussac's Process for assaying Silver Alloys.

THIS *modus operandi* is one which, from its very simplicity and shortness, almost necessarily insures success, but it is only intended for alloys, such as coins, and can, therefore, if applied to ores, only be used to ascertain the real amount of silver in the button produced in the last assay, which, as already observed, contains a very minute amount of spurious metals in an alloyed state.

It was proposed by Gay-Lussac, and from the indubitableness of its results, has since been introduced in France, and most parts of Germany, to regulate the assays of the mint.

They dissolve a given weight of the

alloy in nitric acid and precipitate from the solution all the silver as chloride of silver by means of a standard solution of sodium chloride. For this purpose a *normal solution of common salt* is required, 100 cubic centimeters of which will precipitate 1 gramme of chemically pure silver. There is further required a *decinormal solution of common salt*, containing but one-tenth the quantity of common salt in solution in the first, and a *decinormal solution* of silver in nitric acid containing 1 milligramme of silver in 1 cubic centimeter of solution.

Preparation of the assay solution. The degree of richness of the silver is approximately determined by a preliminary assay, 4 to 6 thousandths parts the amount of silver found by this assay being added to the result. It is generally preferred to assume the degree of richness a few thousandths higher than is actually the case, and to base the calculation for the quantity of assay-sample required upon this, as, to

effect the more rapid settling of the silver chloride, it is more preferable to add, during the titration, a few thousandths from the decinormal solution of salt than to be obliged to add from the decinormal solution of silver. The quantity of alloy containing 1 gramme of silver which is to be taken is then calculated (for instance, if the preliminary assay gives a content of 897 thousandths, then 1.115 grammes of alloy containing 1 gramme of silver should be taken, $1000 : 897 = x : 1000$). The sample in the form of shavings or granules is placed in a numbered flask, together with 6 to 7 cubic centimeters of nitric acid free from chlorine, and dissolved either on a water or sand bath. The flasks in which the samples are dissolved are from 10 to 15 centimeters high, and 5 to $5\frac{1}{2}$ centimeters wide. If several assays are to be made, it is advisable to dip the flasks, which are arranged upon a stand, Fig. D, into hot water. (A black residue may be gold or sulphide of

silver; should the latter be the case, some concentrated nitric acid is added and the fluid heated, or sulphuric acid used.) The nitrous acid formed is then driven out of the flask by means of a small bellows with curved extremity, and the contents of the flask are treated with the normal solution.

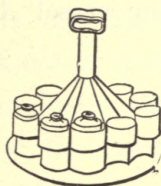


FIG. D.

But, as the influence of the temperature upon the volume of the normal solution of common salt must be taken into consideration, its titer must always be determined on the same day the assays are to be made, with 1 gramme of pure silver + 1 to 2 cubic centimeters decinormal solution of silver, in order to be able, for the above-mentioned reason, to use decinormal solution of salt for the final titration. The silver solution is then titrated by placing the glass flask in the metal cylinder *C*, Fig. E, standing upon the sliding carriage *B* (Sire's apparatus). The glass-cock *c* (a pinch-cock may be used

instead) is then opened and, accompanied by the admission of air through *a*, the normal solution of sodium chloride flows from the vessel *A* through *h*, the thermometer

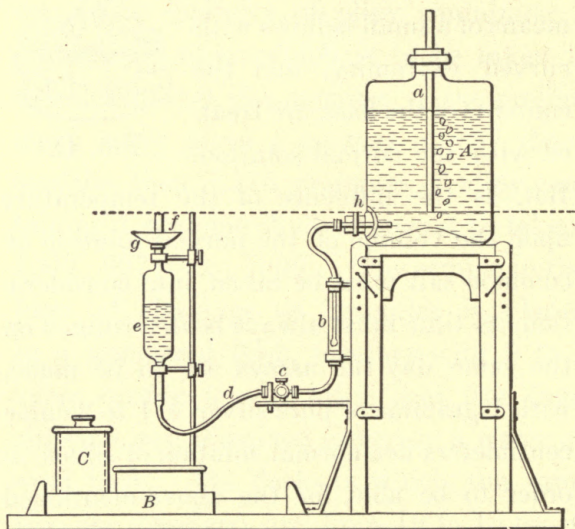


FIG. E.

tube *b*, and the rubber tube *d*, into the burette *e*. It ascends in this, and a small quantity reaches the saucer *g* through the orifice *f*. The cock *c* is now closed (*h* and

e may be also directly connected by a rubber tube provided with a clip) and the pipette *e*, which is now filled, will contain exactly 100 cubic centimeters of liquid. The index finger of the left hand is now placed upon the mouth *f* of the pipette, the rubber tube *d* is detached from the lower end of the pipette *e*, and the sliding carriage *B*, upon which stands the metal cylinder *C* containing the flask with the solution of silver, is pushed underneath the discharge orifice of the pipette. The index finger is now removed from *f*, and the 100 cubic centimeters of the common salt solution are allowed to run into the flask, care being taken that the pipette does not rest on the neck of the flask. The sliding carriage is then pushed back, the flask is closed with its ground-glass stopper, and its contents are cleared by shaking, which is best done by inclosing it for the purpose in a metal cylinder of proper size. If many

assays are to be made, it is advisable to use Gay-Lussac's or Mulder's agitator.

One cubic centimeter of decinormal solution is now added to the entirely clear fluid standing over the precipitate of silver chloride, by means of a graduated pipette contained in a flask, Fig. F, whereby the point of the pipette should be placed against the neck of the flask containing the silver



solution. If turbidity is produced, the silver solution is agitated until it is again clear, and 1 cubic centimeter of the decinormal solution of common salt again added, etc., until the last cubic centimeter which is added does not produce any turbidity. This last cubic centimeter is not taken into calculation, and only one-half of the one previously added. (For the reason previously stated, it is more suitable to use decinormal solution of common salt than decinormal solution of silver for the final reaction.)

Calculation. Suppose the richness of

the alloy was found according to the preliminary assay to be 897 thousandths, 1115 thousandths of the sample containing 1.000 gramme would have to be weighed off. 1000 cubic centimeters of the decinormal solution of salt = 1 gramme of silver. Now suppose 1002.5 cubic centimeters of decinormal solution of common salt had been used, 1000 parts of the alloy would therefore contain 899.1 parts of silver.

In case *mercury* should be present, sodium acetate (0.5 gramme to 5 thousandths of mercury) is added, which will prevent the mercury from being precipitated by the sodium chloride solution; or the mercury is previously volatilized by heating the assay sample in a small graphite crucible in the muffle. For *bismuth* some tartaric acid is added. In case *tin* is present, the assay sample is dissolved in sulphuric acid instead of nitric acid. According to Thorpe, only 2 parts of silver chloride freshly precipitated, and 0.8 part that has been blackened

by exposure to light, are dissolved in 106,000 parts of nitric acid.

Preparation of the normal solutions. A completely saturated solution of common salt is prepared, of which, if the salt used is entirely pure, 170 cubic centimeters contain 54.15 grammes of common salt. These 170 centimeters are diluted to the volume of 10 liters. 100 cubic centimeters of this solution correspond to 0.5415 gramme of common salt, which will completely precipitate 1 gramme of pure silver. The true standard is obtained by pouring 100 cubic centimeters of the common salt solution into a solution of 1 gramme of chemically pure silver. This is agitated by shaking until it becomes clear, and the number of thousandths of common salt or silver which remain free are exactly determined by the addition of an observed volume of very dilute salt solution of known strength, or of a decinormal solution of silver, and from this the quantity of water or of common

salt is calculated which must be added to obtain the correct standard. When this addition has been made, a new test is had with the standard solution and the decinormal solution prepared from it, and this is continued until the solution does not show a perceptible variation from the correct standard

The decinormal solution of common salt is prepared by pouring 100 cubic centimeters of the standard common salt solution into a flask capable of holding 1 liter and filling it with water to the liter mark. For the decinormal solution of silver 1 gramme of fine silver is dissolved in 5 to 6 grammes of nitric acid, which is then diluted with water to 1 liter.

When a large number and great variety of silver alloys have to be assayed, the following tables, A and B, calculated by Gay-Lussac for his silver assay, will be found to save much time and trouble in determining the fineness of the alloy. If,

after the addition of the standard common salt solution, no reaction takes place on adding the decimal salt solution or the decimal silver solution, it is an indication that the silver in the alloy assayed amounts to exactly 1000 milligrammes, and it further indicates that the standard solution is correct. The titer is indicated in the columns by the figure 0.

Table A.—For decimal salt solution.

| Weight of alloy assayed in milli- grammes. | Cubic centimeters of decinormal salt solution added. | | | | | | | | | |
|---|--|-------|-------|--------|-------|--------|-------|-------|-------|--------|
| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 10 |
| correspond to a content of fine silver of | | | | | | | | | | |
| 1000 | 1000.0 | — | — | — | — | — | — | — | — | — |
| 1003 | 997.0 | 998.0 | — | 1000.0 | — | — | — | — | — | — |
| 1005 | 995.0 | 996.0 | 997.0 | 998.0 | 999.0 | 1000.0 | — | — | — | — |
| 1007 | 993.0 | 994.0 | 995.0 | 996.0 | 997.0 | 998.0 | 999.0 | — | — | — |
| 1009 | 991.0 | 992.0 | 993.0 | 994.0 | 995.0 | 996.0 | 997.0 | 998.0 | 999.0 | 1000.0 |
| 1010 | 990.1 | 991.1 | 992.1 | 993.1 | 994.1 | 995.0 | 996.0 | 997.0 | 998.0 | 999.0 |
| 1011 | 989.1 | 990.1 | 991.1 | 992.1 | 993.1 | 994.1 | 995.1 | 996.0 | 997.0 | 998.0 |
| 1015 | 985.2 | 986.2 | 987.2 | 988.2 | 989.2 | 990.1 | 991.1 | 992.1 | 993.1 | 994.1 |
| 1020 | 980.4 | 981.4 | 982.4 | 983.3 | 984.3 | 985.3 | 986.3 | 987.2 | 988.2 | 989.2 |
| 1025 | 975.6 | 976.6 | 977.6 | 978.5 | 979.5 | 980.5 | 981.5 | 982.4 | 983.4 | 984.4 |
| 1030 | 970.9 | 971.8 | 972.8 | 973.8 | 974.8 | 975.7 | 976.7 | 977.7 | 978.6 | 979.6 |
| 1035 | 966.2 | 967.1 | 968.1 | 969.1 | 970.0 | 971.0 | 972.0 | 972.9 | 973.9 | 974.9 |
| 1040 | 961.5 | 962.5 | 963.5 | 964.4 | 965.4 | 966.3 | 967.3 | 968.3 | 969.2 | 970.2 |
| 1045 | 956.9 | 957.9 | 958.8 | 959.8 | 960.8 | 961.7 | 962.7 | 963.6 | 964.6 | 965.5 |
| 1050 | 952.4 | 953.3 | 954.3 | 955.2 | 956.2 | 957.1 | 958.1 | 959.0 | 960.0 | 960.9 |
| 1055 | 947.9 | 948.8 | 949.8 | 950.7 | 951.7 | 952.6 | 953.5 | 954.5 | 955.4 | 956.4 |
| 1060 | 943.4 | 944.3 | 945.3 | 946.2 | 947.2 | 948.1 | 949.1 | 950.0 | 950.9 | 951.9 |
| 1065 | 939.0 | 939.9 | 940.8 | 941.8 | 942.7 | 943.7 | 944.6 | 945.5 | 946.5 | 947.4 |
| 1070 | 934.6 | 935.5 | 936.4 | 937.4 | 938.3 | 939.3 | 940.2 | 941.1 | 942.1 | 943.0 |
| 1075 | 930.2 | 931.2 | 932.1 | 933.0 | 933.9 | 934.9 | 935.8 | 936.7 | 937.7 | 938.6 |

Table A.—For decimal salt solution—(Continued).

| Weight of alloy assayed in milli- grammes. | Cubic centimeters of decinormal salt solution added. | | | | | | | | | | |
|---|--|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| | correspond to a content of fine silver of | | | | | | | | | | |
| 1080 | 925.9 | 926.8 | 927.8 | 928.7 | 929.6 | 930.6 | 931.5 | 932.4 | 933.3 | 934.3 | 935.2 |
| 1085 | 921.7 | 922.6 | 923.5 | 924.4 | 925.3 | 926.3 | 927.2 | 928.1 | 929.0 | 930.0 | 930.9 |
| 1090 | 917.4 | 918.3 | 919.3 | 920.2 | 921.1 | 922.0 | 922.9 | 923.8 | 924.8 | 925.7 | 926.6 |
| 1095 | 913.2 | 914.2 | 915.1 | 916.0 | 917.0 | 917.8 | 918.7 | 919.6 | 920.5 | 921.5 | 922.4 |
| 1100 | 909.1 | 910.0 | 910.9 | 911.8 | 912.7 | 913.6 | 914.5 | 915.4 | 916.4 | 917.3 | 918.2 |
| 1105 | 905.0 | 905.9 | 906.8 | 907.7 | 908.6 | 909.5 | 910.4 | 911.3 | 912.2 | 913.1 | 914.0 |
| 1110 | 900.9 | 901.8 | 902.7 | 903.6 | 904.5 | 905.4 | 906.3 | 907.2 | 908.1 | 909.0 | 909.9 |
| 1115 | 896.9 | 897.8 | 898.6 | 899.5 | 900.4 | 901.3 | 902.2 | 903.1 | 904.0 | 904.9 | 905.8 |
| 1120 | 892.9 | 893.7 | 894.6 | 895.5 | 896.4 | 897.3 | 898.2 | 899.1 | 900.0 | 900.9 | 901.8 |
| 1125 | 888.9 | 889.8 | 890.7 | 891.6 | 892.4 | 893.3 | 894.2 | 895.1 | 896.0 | 896.9 | 897.8 |
| 1130 | 885.0 | 885.8 | 886.7 | 887.6 | 888.5 | 889.4 | 890.3 | 891.2 | 892.0 | 892.9 | 893.8 |
| 1135 | 881.1 | 881.9 | 882.8 | 883.7 | 884.6 | 885.5 | 886.3 | 887.2 | 888.1 | 889.0 | 889.9 |
| 1140 | 877.2 | 878.1 | 878.9 | 879.8 | 880.7 | 881.6 | 882.5 | 883.3 | 884.2 | 885.1 | 886.0 |
| 1145 | 873.4 | 874.2 | 875.1 | 876.0 | 876.9 | 877.7 | 878.6 | 879.5 | 880.3 | 881.2 | 882.1 |
| 1150 | 869.6 | 870.4 | 871.3 | 872.2 | 873.0 | 873.9 | 874.8 | 875.7 | 876.5 | 877.4 | 878.3 |
| 1155 | 865.8 | 866.7 | 867.5 | 868.4 | 869.3 | 870.1 | 871.0 | 871.9 | 872.7 | 873.6 | 874.5 |
| 1160 | 862.1 | 862.9 | 863.8 | 864.7 | 865.5 | 866.4 | 867.2 | 868.1 | 869.0 | 869.8 | 870.7 |
| 1165 | 858.4 | 859.2 | 860.1 | 860.9 | 861.8 | 862.7 | 863.5 | 864.4 | 865.2 | 866.1 | 866.9 |
| 1170 | 854.7 | 855.6 | 856.4 | 857.3 | 858.1 | 859.0 | 859.8 | 860.7 | 861.5 | 862.4 | 863.2 |
| 1175 | 851.1 | 851.9 | 852.8 | 853.6 | 854.5 | 855.3 | 856.2 | 857.0 | 857.9 | 858.7 | 859.6 |

Table A.—For decimal salt solution—(Continued).

| Weight of alloy assayed, in milli- grammes. | Cubic centimeters of decinormal salt solution added. | | | | | | | | | |
|--|--|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 10 |
| | correspond to a content of fine silver of | | | | | | | | | |
| 1180 | 847.5 | 848.3 | 849.2 | 850.0 | 850.8 | 851.7 | 852.5 | 853.4 | 854.2 | 855.1 |
| 1185 | 843.9 | 844.7 | 845.6 | 846.4 | 847.3 | 848.1 | 848.9 | 849.8 | 850.6 | 851.5 |
| 1190 | 840.3 | 841.2 | 842.0 | 842.9 | 843.7 | 844.5 | 845.4 | 846.2 | 847.1 | 847.9 |
| 1195 | 836.8 | 837.7 | 838.5 | 839.3 | 840.2 | 841.0 | 841.8 | 842.7 | 843.5 | 844.3 |
| 1200 | 833.3 | 834.2 | 835.0 | 835.8 | 836.7 | 837.5 | 838.3 | 839.2 | 840.0 | 840.8 |
| 1205 | 829.9 | 830.7 | 831.5 | 832.4 | 833.2 | 834.0 | 834.8 | 835.7 | 836.5 | 837.3 |
| 1210 | 826.4 | 827.3 | 828.1 | 828.9 | 829.7 | 830.6 | 831.4 | 832.2 | 833.1 | 833.9 |
| 1215 | 823.0 | 823.9 | 824.7 | 825.5 | 826.3 | 827.2 | 828.0 | 828.8 | 829.6 | 830.4 |
| 1220 | 819.7 | 820.5 | 821.3 | 822.1 | 822.9 | 823.8 | 824.6 | 825.4 | 826.2 | 827.0 |
| 1225 | 816.3 | 817.1 | 818.0 | 818.8 | 819.6 | 820.4 | 821.2 | 822.0 | 822.9 | 823.7 |
| 1230 | 813.0 | 813.8 | 814.6 | 815.4 | 816.3 | 817.1 | 817.9 | 818.7 | 819.5 | 820.3 |
| 1235 | 809.7 | 810.5 | 811.3 | 812.1 | 813.0 | 813.8 | 814.6 | 815.4 | 816.2 | 817.0 |
| 1240 | 806.5 | 807.3 | 808.1 | 808.9 | 809.7 | 810.5 | 811.3 | 812.1 | 812.9 | 813.7 |
| 1245 | 803.2 | 804.0 | 804.8 | 805.6 | 806.4 | 807.2 | 808.0 | 808.8 | 809.6 | 810.4 |
| 1250 | 800.0 | 800.8 | 801.6 | 802.4 | 803.2 | 804.0 | 804.8 | 805.6 | 806.4 | 807.2 |
| 1255 | 796.8 | 797.6 | 798.4 | 799.2 | 800.0 | 800.8 | 801.6 | 802.4 | 803.2 | 804.0 |

Table B.—For decinormal silver solution.

| Weight of alloy assayed, in milli- grammes. | Cubic centimeters of decinormal silver solution added. | | | | | | | | | | |
|--|--|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| correspond to a content of fine silver of | | | | | | | | | | | |
| 1000 | 1000.0 | 999.0 | 998.0 | 997.0 | 996.0 | 995.0 | 994.0 | 993.0 | 992.0 | 991.0 | 990.0 |
| 1003 | 997.0 | 996.0 | 995.0 | 994.0 | 993.0 | 992.0 | 991.0 | 990.0 | 989.0 | 988.0 | 987.0 |
| 1005 | 995.0 | 994.0 | 993.0 | 992.0 | 991.0 | 990.0 | 989.0 | 988.1 | 987.1 | 986.1 | 985.1 |
| 1007 | 993.0 | 992.0 | 991.1 | 990.1 | 989.1 | 988.1 | 987.1 | 986.1 | 985.1 | 984.1 | 983.1 |
| 1009 | 991.0 | 990.0 | 989.0 | 988.0 | 987.0 | 986.0 | 985.0 | 984.0 | 983.0 | 982.0 | 981.0 |
| 1010 | 990.1 | 989.1 | 988.1 | 987.1 | 986.1 | 985.1 | 984.2 | 983.2 | 982.2 | 981.2 | 980.2 |
| 1011 | 989.1 | 988.1 | 987.1 | 986.2 | 985.2 | 984.2 | 983.2 | 982.2 | 981.2 | 980.2 | 979.2 |
| 1015 | 985.2 | 984.2 | 983.2 | 982.3 | 981.3 | 980.3 | 979.3 | 978.3 | 977.3 | 976.4 | 975.4 |
| 1020 | 980.4 | 979.4 | 978.4 | 977.4 | 976.5 | 975.5 | 974.5 | 973.5 | 972.5 | 971.6 | 970.6 |
| 1025 | 975.6 | 974.6 | 973.7 | 972.7 | 971.7 | 970.7 | 969.8 | 968.8 | 967.8 | 966.8 | 965.8 |
| 1030 | 970.9 | 969.9 | 968.9 | 968.0 | 967.0 | 966.0 | 965.0 | 964.1 | 963.1 | 962.1 | 961.2 |
| 1035 | 966.2 | 965.2 | 964.2 | 963.3 | 962.3 | 961.3 | 960.4 | 959.4 | 958.4 | 957.5 | 956.5 |
| 1040 | 961.5 | 960.6 | 959.6 | 958.6 | 957.7 | 956.7 | 955.8 | 954.8 | 953.8 | 952.9 | 951.9 |
| 1045 | 956.9 | 956.0 | 955.0 | 954.1 | 953.1 | 952.1 | 951.2 | 950.2 | 949.3 | 948.3 | 947.4 |
| 1050 | 952.4 | 951.4 | 950.5 | 949.5 | 948.6 | 947.6 | 946.7 | 945.7 | 944.8 | 943.8 | 942.9 |
| 1055 | 947.9 | 946.9 | 946.0 | 945.0 | 944.1 | 943.1 | 942.2 | 941.2 | 940.3 | 939.3 | 938.4 |
| 1060 | 943.4 | 942.4 | 941.5 | 940.6 | 939.6 | 938.7 | 937.7 | 936.8 | 935.8 | 934.9 | 934.0 |
| 1065 | 939.0 | 938.0 | 937.1 | 936.1 | 935.2 | 934.3 | 933.3 | 932.4 | 931.4 | 930.5 | 929.6 |
| 1070 | 934.6 | 933.6 | 932.7 | 931.8 | 930.8 | 929.9 | 929.0 | 928.0 | 927.1 | 926.2 | 925.2 |
| 1075 | 930.2 | 929.3 | 928.4 | 927.4 | 926.5 | 925.6 | 924.7 | 923.7 | 922.8 | 921.9 | 920.9 |

Table B.—For decimal silver solution—(Continued).

| Weight of alloy assayed, in milli- grammes. | Cubic centimeters of decimal silver solution added. | | | | | | | | | |
|--|---|-------|-------|-------|-------|-------|-------|-------|-------|-------------|
| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 10 |
| | correspond to a content of fine silver of | | | | | | | | | |
| 1080 | 925.9 | 925.0 | 924.1 | 923.1 | 922.2 | 921.3 | 920.4 | 919.4 | 918.5 | 917.6 916.7 |
| 1085 | 921.7 | 920.7 | 919.8 | 918.9 | 918.0 | 917.0 | 916.1 | 915.2 | 914.3 | 913.4 912.4 |
| 1090 | 917.4 | 916.5 | 915.6 | 914.7 | 913.8 | 912.8 | 911.9 | 911.0 | 910.1 | 909.2 908.3 |
| 1095 | 913.2 | 912.3 | 911.4 | 910.5 | 909.6 | 908.7 | 907.8 | 906.8 | 905.9 | 905.0 904.1 |
| 1100 | 909.1 | 908.2 | 907.3 | 906.4 | 905.4 | 904.5 | 903.6 | 902.7 | 901.8 | 900.9 900.0 |
| 1105 | 905.0 | 904.1 | 903.2 | 902.3 | 901.4 | 900.4 | 899.5 | 898.6 | 897.7 | 896.8 895.9 |
| 1110 | 900.9 | 900.0 | 899.1 | 898.2 | 897.3 | 896.4 | 895.5 | 894.6 | 893.7 | 892.8 891.9 |
| 1115 | 896.9 | 896.0 | 895.1 | 894.2 | 893.3 | 892.4 | 891.5 | 890.6 | 889.7 | 888.8 887.9 |
| 1120 | 892.9 | 892.0 | 891.1 | 890.2 | 889.3 | 888.4 | 887.5 | 886.6 | 885.7 | 884.8 883.9 |
| 1125 | 888.9 | 888.0 | 887.1 | 886.2 | 885.3 | 884.4 | 883.6 | 882.7 | 881.8 | 880.9 880.0 |
| 1130 | 885.0 | 884.1 | 883.2 | 882.3 | 881.4 | 880.5 | 879.6 | 878.8 | 877.9 | 877.0 876.1 |
| 1135 | 881.1 | 880.2 | 879.3 | 878.4 | 877.5 | 876.7 | 875.8 | 874.9 | 874.0 | 873.1 872.3 |
| 1140 | 877.2 | 876.3 | 875.4 | 874.6 | 873.7 | 872.8 | 871.9 | 871.0 | 870.2 | 869.3 868.4 |
| 1145 | 873.4 | 872.5 | 871.6 | 870.7 | 869.9 | 869.0 | 868.1 | 867.2 | 866.4 | 865.5 864.6 |
| 1150 | 869.6 | 868.7 | 867.8 | 867.0 | 866.1 | 865.2 | 864.3 | 863.5 | 862.6 | 861.7 860.9 |
| 1155 | 865.8 | 864.9 | 864.1 | 863.2 | 862.3 | 861.5 | 860.6 | 859.7 | 858.9 | 858.0 857.1 |
| 1160 | 862.1 | 861.2 | 860.3 | 859.5 | 858.6 | 857.8 | 856.9 | 856.0 | 855.2 | 854.3 853.4 |
| 1165 | 858.4 | 857.5 | 856.6 | 855.8 | 854.9 | 854.1 | 853.2 | 852.4 | 851.5 | 850.6 849.8 |
| 1170 | 854.7 | 853.8 | 853.0 | 852.1 | 851.3 | 850.4 | 849.6 | 848.7 | 847.9 | 847.0 846.1 |
| 1175 | 851.1 | 850.2 | 849.4 | 848.5 | 847.7 | 846.8 | 846.0 | 845.1 | 844.3 | 843.4 842.5 |

Table B.—For decinormal silver solution—(Continued).

| Weight of alloy assayed, in milli- grammes. | Cubic centimeters of decinormal silver solution added. | | | | | | | | | | |
|--|--|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| | correspond to a content of fine silver of | | | | | | | | | | |
| 1180 | 847.5 | 846.6 | 845.8 | 844.9 | 844.1 | 843.2 | 842.4 | 841.5 | 840.7 | 839.8 | 839.0 |
| 1185 | 843.9 | 843.0 | 842.2 | 841.3 | 840.5 | 839.7 | 838.8 | 838.0 | 837.1 | 836.3 | 835.4 |
| 1190 | 840.3 | 839.5 | 838.7 | 837.8 | 837.0 | 836.1 | 835.3 | 834.5 | 833.6 | 832.8 | 831.9 |
| 1195 | 836.8 | 836.0 | 835.1 | 834.3 | 833.5 | 832.6 | 831.8 | 831.0 | 830.1 | 829.3 | 828.4 |
| 1200 | 833.3 | 832.5 | 831.7 | 830.8 | 830.0 | 829.2 | 828.3 | 827.5 | 826.7 | 825.8 | 825.0 |
| 1205 | 829.9 | 829.0 | 828.2 | 827.4 | 826.6 | 825.7 | 824.9 | 824.1 | 823.2 | 822.4 | 821.6 |
| 1210 | 826.4 | 825.6 | 824.8 | 824.0 | 823.1 | 822.3 | 821.5 | 820.7 | 819.8 | 819.0 | 818.2 |
| 1215 | 823.0 | 822.2 | 821.4 | 820.6 | 819.7 | 818.9 | 818.1 | 817.3 | 816.5 | 815.6 | 814.8 |
| 1220 | 819.7 | 818.8 | 818.0 | 817.2 | 816.4 | 815.6 | 814.7 | 813.9 | 813.1 | 812.3 | 811.5 |
| 1225 | 816.3 | 815.5 | 814.7 | 813.9 | 813.1 | 812.2 | 811.4 | 810.6 | 809.8 | 809.0 | 808.2 |
| 1230 | 813.0 | 812.2 | 811.4 | 810.6 | 809.8 | 808.9 | 808.1 | 807.3 | 806.5 | 805.7 | 804.9 |
| 1235 | 809.7 | 808.9 | 808.1 | 807.3 | 806.5 | 805.7 | 804.9 | 804.0 | 803.2 | 802.4 | 801.6 |
| 1240 | 806.5 | 805.6 | 804.8 | 804.0 | 803.2 | 802.4 | 801.6 | 800.8 | 800.0 | 799.2 | 798.4 |
| 1245 | 803.2 | 802.4 | 801.6 | 800.8 | 800.0 | 799.2 | 798.4 | 797.6 | 796.8 | 796.0 | 795.2 |
| 1250 | 800.0 | 799.2 | 798.4 | 797.6 | 796.8 | 796.0 | 795.2 | 794.4 | 793.6 | 792.8 | 792.0 |
| 1255 | 796.8 | 796.0 | 795.2 | 794.4 | 793.6 | 792.8 | 792.0 | 791.2 | 790.4 | 789.6 | 788.8 |

In making Gay-Lussac's assay of silver bullion, a great deal of time is necessarily spent in waiting for the suspended chloride to settle and leave the liquid clear in order to observe the action of the next drop of the precipitant. Whittell has reduced this loss of time and insured greater facility in making the assay by dividing the solution containing the silver into several, say five, equal parts in separate vessels. They are placed in a row, and 3 cubic centimeters of salt solution added to the first, 4 c. c. to the second, 5 c. c. to the next, and so on. After the precipitate has subsided, $\frac{1}{2}$ c. c. of the same solution is added to each successively. Numbers 1, 2, and 3 will perhaps show traces of silver still in solution, but numbers 4 and 5 none. The amount precipitated from number 3 multiplied by 5 (as it represents only $\frac{1}{5}$ of the original solution of silver) will be the amount of silver contained in the ore or alloy being assayed. A simple

means of settling the precipitated chloride almost instantaneously is to agitate the solution with a few drops of chloroform. Its action seems to be entirely mechanical. The agitation displaces the chloroform in minute globules throughout the silver solution, which in settling to the bottom carries with it every particle of the chloride.

VI.

Assay of Native Silver Ores.

THE following method is recommended by Lowe. When silver is chemically combined in the ore it is not difficult to determine its assay value, but in ores containing the silver in a metallic state, the assay can yield only approximate results. The following method obviates this difficulty to some extent. The ore is coarsely powdered and sampled down to a half pound, then finely powdered and passed through a 100 mesh sieve. The sifted ore is divided into four parts and four assays made of it, each amount for each assay being taken as nearly as possible from the same relative parts of each quarter. The average of the four assays is taken as the result. The metallic par-

ticles or scales of silver which will not pass through the meshes of the sieve are mixed with assay lead, cupelled, and the weight of the resulting button reduced to its proper value added to the result obtained from the assay of the siftings.

VII.

Assay of Gold Ores by Heat.

THOSE properties of silver, which enable us to assay its ores by the process of cupellation, are so equally characteristic of gold and its ores, that, were I to dwell more at length on this first part of the operation, it would be a mere repetition of what has been said under the head of Silver, and therefore I shall only refer the reader to the remarks given above.

When, however, that process is completed, and when therefore with silver we would have procured the final result, the real gold assay only commences, for gold, though always native, contains silver, platinum, &c.

I must remark that, since gold ores are almost invariably excessively poor, com-

pared with those of other metals, we should arrange our assays accordingly, and commence with a larger amount of ore than is necessary in other cases. For this purpose it is common to begin with six different parts of ore of 50 grs. each, which, after being mixed with lead and borax as with silver, are committed to the muffle in as many separate crucibles. The products are then melted up into two buttons, in two different cupels of the same kind, and these again in the process of extracting the lead are concentrated into one. Of course, when the assay is finished we are obliged to divide the produce by six to ascertain the per-centage amount of gold in the specimen under examination.

After having proceeded thus far, it is necessary to procure some perfectly pure silver, for which we have the following methods: first, by producing chloride of silver, which is done by precipitating the silver from the nitric acid solution, with muriatic acid, and

melting it in a porcelain crucible, with potash; chloride of potash and the pure silver being thus formed: or secondly by the galvanic process, the virgin metal being thus collected round a rod of zinc immersed in the acid (nitric) liquid from which, as above, the silver has been extracted by adding muriatic acid, but which precipitate must remain in the same vessel. The silver is then preserved in the shape of filings.

It is necessary to make use of this silver whenever the quantity of gold in the last produced button is more than one fourth, (hence the term *quartation*) since experience has shown that if there are not at least about three quarters the amount of silver (rarely the case with ores) in this alloy, the gold would protect the particles of silver from the action of the acid, which, as will become evident from the sequel, would prove fatal to our investigations.

Of the pure silver, two and a half times

the weight of the buttons of impure gold, and about half the weight of both in pure lead, should be melted up with them in one crucible, (fig. 2, Plate II.) This single button of gold and silver alloy is to be hammered flat on a little steel anvil, and slightly curved, as this shape will allow it more easily to pass through the mouth of a little vial, in which it is now placed to be boiled with nitric acid (2 drams) of from 1 to 3 specific gravity. The vial should be round at the bottom as this is performed over a spirit-lamp. We allow the liquid to boil until the yellowish vapours of nitrous acid gas have ceased to be emitted. The gold is then pure, and requires only to be boiled a couple of times in water to remove the acid, and then to be heated to redness to evaporate the water, to enable us to weigh it, which, as with all other assays, should never be done till the metal is perfectly cold.

If a small portion of platinum be in the ore, it will be found in the nitric acid solution of silver. (See Platinum.)

The original amount of silver may be ascertained by deducting the weight of the gold from the button produced before the other pure silver was added.

VIII.

Assay of Gold Ores by Amalgamation.

THIS method is founded on the fact, that gold unites with quicksilver to form an amalgam, for thus the alloys of mercury and other metals are termed. It is one that may answer for peculiar circumstances, though it is not accurate enough to serve as a regular dokimastic* test, since lead and silver also produce amalgams.

We require pure mercury for this purpose; and as the quicksilver of commerce generally contains some lead, we must purify it by pressing it through a piece of buckskin.

The powdered gold ore, which ought

* A technical term, derived from the Greek verb δοκιμάζειν, to try, to test the purity of a thing.

first to be reduced in volume by washing, is brought in contact with the quicksilver, the sand or gangue-rock removed by sifting the mass through a coarse cloth; after which, by forcing the quicksilver through some buckskin, the amalgam is retained.

By evaporating the mercury in an iron spoon over a lamp, the gold is procured, which will, however, still be found to contain silver or lead, if these occur in the ore.

IX.

Assay of Gold by Washing.

THIS is a way of ascertaining the approximate value of a gold ore, which hardly deserves being called an assay, although, with an experienced hand, tolerable accuracy may be attained.

It is based on the fact, that gold is much heavier than quartz or other ganguerock, and therefore the rock should be well pounded for this operation.

The process is precisely the same as that used to extract gold from the sand of river beds, and which is now so extensively employed in California. Even the vessels used are frequently of the same kind, though it is better to have a small glass cylinder, about three times the length of the part that will be occupied by the ore,

and three-quarters of an inch in diameter, closed at the end, like a common chemical test-glass. I would suggest, if, where used, the gangue-rock or sand be always of the same kind, having a mark round the tube made with a slight scratch of the diamond, up to where a certain quantity by weight of the ore would reach. One hundred grains would not be too much, if, for instance, the rock is quartz; and it is also the most convenient amount, from the facility thus afforded in afterward calculating the per-centage. Under circumstances where we have to deal with ores in which the original rock is not always the same, it would be well to have several such tubes, to each of which its peculiar rock might be allotted; or one with differently marked lines. In this manner, one such line might indicate the part of the tube which would be filled by a hundred grains of the quartzose ore, another the portion which would be occupied by

the same weight of a talcose one, and so on. The lines will vary in height as the ratio of the various specific gravities of the respective ores.

When the properly pulverized ore has been placed in the glass tube, the latter should be filled up two-thirds with water, and tightly corked; after which, by repeated shakings, the gold will be collected below. By careful decanting and continual shaking, we can remove the sand and particles of rock, and retain the gold as a matter of course, though only in its natural, alloyed state.

This method will be found to be a considerable improvement on the washing in pans. If the ore contains iron pyrites, it is best to calcine it first, though carefully, so that no gold is carried off mechanically by the gases formed.

X.

Assay of Gold by Panning.

THE pan-test is a process of concentration, the product being either gold particles, or gold sulphides, iron, sand, etc., depending on how far the process is carried.

The pan used for the purpose is a sheet-iron vessel of a shallow truncated conical shape, or a round shallow wooden dish with its bottom sloping to a point and technically known as a "batea." In the United States experienced miners frequently use a section of a bullock's horn as a substitute for the pan.

The ore being sampled, crushed and pulverized so as to pass through a 50 or 60 mesh sieve, the requisite amount (3 to 15 kilogrammes) is weighed out and put in the pan, which must be free from grease.

Now moisten the ore and let it stand for a few moments, in order that particles may not float off when the pan is put in water. When wet the whole pan and ore is gently sunk below the surface of a tank of water and a peculiar oscillatory motion or side vibration is commenced, though not enough to throw any particles of ore over the edges of the pan. The object of this is to settle the heavier particles (the free gold, heavy minerals, black sand, etc.), and have nothing on the surface but rock or quartz. Then slightly incline the pan, and so wash it around as to carry the surface rock over the edge; only, however, a little at a time. Level the pan and resettle as at first; again incline and wash more over the edge. Keep up this operation, gradually getting more and more rock over the edge, and becoming more careful and washing more delicately as the process continues.

Toward the end of the operation, that is, when the rock is nearly gone, be careful to

keep the ore under the surface of the water, as the gold might otherwise become dry and float off. Also make no sudden or unusual lurch, or the whole result may go off the pan. Dry the residue. If gold alone is obtained, that is, gold (or gold and silver) free from sulphides, etc., it must be treated as an alloy, weighed, parted, and weighed again, or cupelled with lead, weighed, parted and weighed; in both cases giving gold and silver. If the panning is not carried to such a point as to get rid of all the rock, the concentration is all scorified with test lead (or melted down in a crucible), cupelled, parted and weighed. In the case of an ore supposed to carry auriferous sulphides, it should be panned so far as can safely be done without losing metalliferous particles, and the concentration treated as above described.

If the ore is quite poor, or a large quantity is desired to be worked, the panning

can be carried on roughly and the successive concentrations finally panned together.

The results are based upon the amount of ore taken in the pan. As an example of the calculation, the following is given :

Weight of panful of ore, $2\frac{1}{4}$ kilogrammes
 $= 2,250,000$ milligrammes.

Weight of bead obtained, gold, 20 milligrammes ; silver, 50 milligrammes ;

$$\text{then } \frac{2,250,000 \text{ mgrms.} \dots 29166}{20 \text{ mgrms.}} \times \frac{x}{100 \text{ oz.}} = 25$$

$$\text{and } \frac{2,250,000 \text{ mgrms.} \dots 29166}{50 \text{ mgrms.}} \times \frac{x}{100 \text{ oz.}} = 64$$

The free gold can be separated from the sulphides (if it is desired to determine how much of the gold is "free" and how much in the "sulphides") by washing in an amalgamated pan. Such a vessel may be simply made by bending a piece of thin silver-plated copper (about 6x12 inches) so as to form curved edges on three sides, the silvered side in. The side not turned up is one of the narrow ends. A little mercury

will quickly amalgamate the interior, and if the ore is carefully washed over this, most of the free gold will become amalgamated and stick to the pan. A piece of chamois skin made into a rubber will push the gold, which can be seen as little specks of amalgam, to the open edge of the pan and into a crucible. The mercury can be driven from the gold by heat.

Kleinschmidt gives the following method of assaying gold by panning. Five kilogrammes of the ore to be assayed are sampled, crushed and pulverized so as to pass through a 50 or 60 mesh sieve, any gold remaining upon the sieve being weighed separately. Of the powdered sample passed through the sieve, 500 grammes are put in the pan, moistened with water and mixed with 5 grammes or more of mercury. After standing two hours, washing is slowly commenced and continued, finally, with the addition of potassium cyanide solution. The cleansed amalgam is gently

heated, and the resulting metal, if necessary, cupelled, or the bead parted.

According to another method the pulverized ore is put in a porcelain dish, and for some time mixed with mercury by means of a piece of amalgamated silver sheet. The gold amalgam is then scraped off, and the entire amalgam treated with nitric acid, whereby the gold remains behind.

In Australia 1 kilogramme of gravelly gold ore is dead-roasted, the roasted sample triturated in an iron mortar with water to a stiff paste, and, after adding one tablespoonful of mercury, the mixture is rubbed together. The addition of mercury and trituration are then repeated, and after adding hot water, soda and 5 or 6 spoonfuls of mercury, the whole is rubbed together, washed in an enamelled dish, collected and the amalgam distilled, whereby 80 to 90 per cent. of the gold which can be brought out by the fire-assay are obtained.

XI.

Assay of Gold by Combined Dry and Wet Methods.

THE object of these assays is to fix the gold, which mostly contains silver, on lead (smelting with lead), which, after cupellation, gives a silver-gold button, to be further parted by the wet way (quartation with nitric acid). In smelting with lead earthy admixtures are scorified, and metallic sulphides previously converted into oxides by oxidizing agents (lead oxide, air, saltpetre, etc.).

The gold being very unequally distributed in the ores, special care must be taken to obtain a correct average sample of the ore to be assayed. To illustrate the averaging, take a quantity of ore weighing about fifty pounds, which may be as a

single lump, or, better, the result of the selection of samples across the section of a vein. With a sledge-hammer break up the entire mass into pieces about the size of a walnut, and form of the mass an oblong pile. Then divide the pile in halves by means of a large spatula or piece of heavy sheet brass. Select one of these halves, and break up the pieces constituting it still finer to the size of a hazelnut or less. Mix the pieces, form them into a pile and halve as before. Continue the crushing, mixing and halving until about one pound has finally been sampled down. Instead of halving the piles may be quartered, and two of the diagonally opposite quarters taken as a half.

The next step is to pulverize the sample finally obtained; wet or damp ores must be dried before pulverizing. The latter may be effected by means of an iron mortar and pestle, care being had to prevent loss due to flying particles by loosely wrapping a

piece of linen around the pestle and across the top of the mortar. Then sift the pulverized sample through a tin box-sieve, having 80 to 100 meshes to the linear inch, upon a piece of brown paper or a zinc sifting-pan.

Assays by smelting with lead. According to the nature of the assay-sample (poor or rich, with free gold, with earthy or gravelly admixtures), there is used—

a, *The scorification assay*, which is suitable for every kind of ore. According to the supposed content of gold weigh out for each assay 0.5 to 10 grammes of the sample. With poor material enough assays are made that the gold-silver button resulting from the cupellation of all the lead buttons weighs 0.2 to 0.5 gramme. The powdered ore, according to whether it contains metallic sulphides or only earthy constituents, is charged with four to sixteen times—on an average with eight times—the quantity of granulated lead free from silver. The object of the latter is to absorb the gold pres-

ent, for which it has great affinity. This is most readily effected by the presence of metallic lead in an earthy substance, but with more difficulty in metallic sulphides. The latter must previously, during the oxidizing melting, be oxidized by the lead oxide formed and converted into oxide. Since, however, the different metallic sulphides are more or less readily completely oxidized by lead oxide, a varying addition of lead is, according to their quality, required. Thus, for instance, galena requires four to six times, iron pyrites nine to ten times, zinc blende and copper pyrites ten to fifteen times the quantity of assay-lead of the ore weighed out. If the ore contains only acid earthy constituents (quartz, clay and other silicates), they are dissolved to a slag by the lead oxide. If it contains basic earths (lime, magnesia, etc.) or metallic oxides produced from metallic sulphides in roasting, acid dissolving agents (borax, glass, glass) are required, the quantity of

such agents being dependent on the quantity of the bases to be scorified, and on their refractoriness. Lime and zinc oxide produce special refractoriness, and require up to 50 per cent. or more of the ore weighed out of fluxing agents; oxides of iron and copper from 10 to 15 per cent.

The assay itself is executed as follows: Have ready cleaned a number of the scorifiers. Number or letter each scorifier with reddle, weigh the requisite amount of granulated lead, divide it in halves and transfer one-half to the scorifier. Upon it place the ore and mix by means of a spatula. Pour the remaining half of the lead evenly over the surface of the mixed ore and lead, and over all sprinkle the borax-glass. A charge frequently used is, for instance, 3.75 grammes ore, 30 grammes lead and 0.94 gramme borax-glass. In similar manner charge all other scorifiers.

By means of the scorifier-tongs place the scorifiers in the strongly-heated muffle-

furnace, close the door and augment the heat (fifteen to twenty-five minutes) until the surface of the assays is smooth without showing unmelted portions (period of hot working). In melting the lead sinks to the bottom, absorbing thereby the greater portion of the gold, while the specifically lighter substances (earths, sulphides, borax) pass to the surface, where they at first remain unmelted. With the increase in temperature and the access of air, which should not be entirely excluded, the metallic sulphides are oxidized, partially directly by the air and partially by the lead oxide formed, and the oxides together with the earths melt with lead oxide and borax to a slag. The latter, however, still holds metallic sulphides (oxysulphides) as well as particles of gold enclosed, and accumulates on the edge of the scorifier. The door of the muffle is then opened, thus admitting a current of air, the fire being at the same time decreased. The lead now oxidizes in

larger quantity, and its oxygen passes over to the metallic sulphides in the slag (period of cold working). As soon as the slag covers the entire surface of the bath, the process is finished, because a further access of air would no longer act upon the lead. Finally close the door of the muffle, and increase the heat for a minute or two to fully liquify the slag.

Now remove the scorifiers from the muffle and pour their contents into the scorification-moulds, which should be warm and painted inside with chalk or redde wash. Cover each receptacle of the moulds with its proper scorifier to retain its identification.

Instead of pouring the contents of the scorifiers into scorification-moulds, the lead buttons may be allowed to cool in their scorifiers. In either case, however, when cold, detach the lead buttons from their slags and hammer each button into a clean cube with flattened corners. A content of

silver in the ore collects with the gold in the lead.

The scorifiers when placed in the hot muffle and taken from it must not crack, and should be perfectly smooth in their interior, so that the corrosion of the clay substance by the lead oxide may not exert a disturbing effect.

b. Crucible assay. Melt the assay-sample together with fluxes (potash, soda, borax, etc.), lead oxide and reducing agents (coal dust, colophony, flour, argol), in crucibles, whereby the earthy constituents are scorified by the fluxes and a portion of the lead oxide, while another portion of the lead oxide is converted by the reducing agents into metallic lead which then absorbs the gold (besides the silver). These assays have the advantage of being more accurate than the scorification-assay, because with the availability of a larger quantity of assay-substance (25 to 50 grammes or more) the losses decrease, and furthermore they

are more convenient and cheaper, on account of a larger number of assays (frequently twenty or more) not being required, which has to be taken into consideration, especially with very poor ore. On the other hand, they are chiefly suitable only for earthy ores or ores containing ferric oxide and products containing native gold (gulch gold, dross or sweepings, auriferous slags, etc.). They are less adapted for metallic sulphides, telluride ores, arsenical and antimonial ores. The latter to be converted into oxides require previous roasting, because by melting they are but incompletely decomposed, while in the more suitable scorification process they suffer vigorous oxidation.

a. Melting earthy gold ores or gold ores containing ferric oxide (also dross or sweepings, slags). Into a smooth refractory clay crucible with steep sides bring 10 grammes of borax and 10 grammes of argol, next 20 grammes of litharge free from silver and

gold, gently breathe upon the sides of the crucible and turn the crucible around until it is coated $\frac{2}{3}$ of its height with litharge. Then add 15 grammes of potash and 25 grammes of the assay-substance, mix the whole with a spatula, add a cover of 10 grammes of soda, then one 12 millimeters thick of common salt, and scatter 5 grammes of litharge around the walls of the crucible.

A charge for American gold ore consists, for instance, of 50 grammes ore, 70 grammes anhydrous soda, 100 to 120 grammes litharge, and 6 to 8 grammes coal dust. First mix the ore, litharge and coal dust by themselves and then with the flux, and lay in the crucible a piece of iron wire, in case a few per cent. of sulphur should be present.

Or, mix 500 grammes of very pure gold sand with 300 grammes potash, 200 grammes soda and 50 grammes borax ; scatter upon the mixture 20 grammes of granular lead free from silver, upon this a thin layer

of soda, and finally a cover of common salt.

The crucible thus charged is slowly heated in the air-furnace until no more flames of carbonic oxide come from under the lid and the puffing up of the mass ceases. The heat is then increased for 10 or 15 minutes more to insure complete fusion. The pasty condition of the mass, which is maintained during flaming, promotes the action of the lead upon the gold. The crucible, when cold, is broken up, the lead freed from slag and cupelled. If great accuracy is required, several assays (4 or 5) are made. The lead buttons obtained are cupelled, the metallic grains weighed, and those best agreeing in weight examined as to their content of gold by the wet process.

b. Melting of sulphuretted ores. Ores with a larger content of gold are brought into scorifiers and dead-roasted in the muffle by gently heating them with the access of

air until a sample taken from the muffle shows no odor of sulphurous acid. The slightly slagged mass of an earthy appearance is then broken up with the blade of the spatula, rubbed fine, mixed with coal dust and again heated, with the access of air, in the muffle until the coal dust is burnt.

With poorer ores, of which a larger quantity, for instance, 0.5 kilogramme or more, has to be used, roasting in the muffle is effected in clay boxes, each about 200 millimeters long, 70 to 90 millimeters wide, and 40 to 50 millimeters deep, or over the coal fire of an air furnace by placing over it a clay-lined piece of sheet-iron turned up on the edges, or a clay roasting-plate.

The charging and melting of the roasted assay-sample is effected in the manner above described, but for the better scorification of the larger amount of oxide, the quantity of borax is somewhat increased or some glass added. Another charge is, for

instance, as follows : The roasted sample resulting from 0.5 kilogramme crude ore, 125 to 250 grammes potash-glass or soda-glass, 125 grammes black flux, or 250 grammes potash and 32 grammes flour, then scattered upon it 0.5 kilogramme of grain-lead free from silver, with a protecting cover of common salt. Waste from amalgamation containing pyrites is melted, without being roasted, with fluxes and litharge without reducing agents. By the content of sulphur in the pyrites lead is then reduced from the lead oxide, the former absorbing the gold and silver. If, with very poor substances, the gold alone is to be determined, four assays of 25 grammes each are made, and the four buttons cupelled with the addition of silver, so that the resulting button with a sufficient content of silver can be at once submitted to quartation. Other fluxes for dross or sweepings are, 4 parts potash and 1 part flour, or 13 parts potash and 10 parts soda with small

quantities of borax and saltpetre. Melt for two hours in a Hessian crucible, flatten out the resulting lead button, cut it up, scorify the separate pieces in scorifiers and then concentrate them to one button, which is cupelled and parted. Merrick melts lead buttons, which are too large for direct cupellation, in a capacious Hessian crucible, adds one-half the quantity of the weight of the button of saltpetre, heats to a white heat, stirs with an iron wire, and then takes the crucible from the furnace. If necessary the operation is repeated, and even pure gold can in this manner be obtained.

c. Cupellation of the auriferous lead.
This operation consists in oxidizing the lead of the lead buttons, the litharge formed by the heat being partly absorbed by the cupel and partly driven up the chimney, leaving the gold and silver together as a bead upon the surface of the cupel. Other metals that may have remained in small quantity from the previous operations are also oxidized

and thus removed. The process is the same as for silver (which see) with the exception of a hotter "driving" towards the end of the assay, so that no litharge remains. If the assay-sample is poor, the separate lead buttons are either entirely cupelled or only partially. In the latter case they are wrapped in lead foil and cupelled together. The resulting gold button is then weighed, and, in case it contains silver, the latter is parted by means of nitric acid.

d. Parting the gold from the silver. The button after weighing is flattened a little. For parting the gold from the silver pure nitric acid free from chlorine is used. The silver under certain conditions dissolves in the heated acid, but not the gold. Parting is complete only when at least $2\frac{1}{2}$ parts of silver are present to 1 part of gold. With a smaller content of silver the button possesses the power of resisting the solvent action of nitric acid. The metallic grains resulting from actual gold ores are apt to be

richer in gold than in silver, whilst those from auriferous silver ores, pyrites, etc., generally contain only $\frac{1}{4}$ to $\frac{1}{3}$ gold. Whilst the latter are directly subjected to the treatment with nitric acid, the former are first again cupelled with two and a half times the quantity of silver enfolded in a small piece of sheet-lead. If the gold alone is to be determined, sufficient silver may at once be added when cupelling the auriferous lead button.

To judge whether silver has to be added or not, note,

a. The color of the button. If it is deep yellow, two and a half to three times the quantity of silver will have to be added; if pale yellow, twice the quantity; and, if white, once the quantity or none. *Or,*

β. The behavior of the button towards nitric acid of 1.19 specific gravity when heated in a small flask. If the laminated button thereby breaks up and brown flakes of gold are separated, sufficient silver (at

least more than 4 parts) is present and the operation may be proceeded with. But if the laminated button does not break up and only acquires a dark coloration, it is taken from the fluid, rinsed off, dried, cupelled with two and a half times the quantity of silver and some lead, and parting completed with nitric acid, whereby the gold remains behind either in a pulverulent form if at least 4 parts silver are present, or in a coherent state with $2\frac{1}{2}$ parts silver to 1 part gold. When no more nitrous acid is developed heating is interrupted, the brown lustreless gold allowed to settle and the liquid carefully decanted. The gold is now washed twice by decantation with boiling distilled water. The flask is then entirely filled with cold water and inverted in a clay crucible, or a small porcelain saucer, and when the gold has dropped into the crucible, the flask is carefully withdrawn over the side. The water is then poured off, the gold dried, and the crucible strongly heated, whereby

the powder unites to a coherent mass which is weighed.

To recognize with very poor ores whether the small black specks remaining behind, when treating the button with nitric acid, are gold, allow them to slide from the flask into a small porcelain dish. Then carefully wash them first with water, next with alcohol and ether, and ignite, whereby with a magnifying glass traces of yellow shining metal may be observed.

Or, dissolve the specks, after transferring to a test tube and washing in aqua regia, evaporate to dryness, and strongly heat the tube, whereby thin yellow rings appear, which may be recognized either with the naked eye or with a magnifying glass.

The specks may also be transferred to a glass plate which is placed under a microscope, and after pouring a few drops of aqua regia upon it, the solution is observed, a yellow fluid being formed, in which after evaporation and heating yellow gold may

be recognized. The smallest quantity of chloride of silver of various colors, especially violet, can be recognized in the gold solution. A black speck rubbed in an agate mortar shows a gold color.

XII.

Assay of Gold Ores by a Wet Process.

ALTHOUGH I have tried to make it a point to introduce as few wet processes as possible into this treatise, I now venture to give directions for one which will be found very available to ascertain the true amount of gold, when the gold has been extracted in its natural state by the last-given methods. It is characterized by the ease with which it can be performed.

The residue of alloyed gold produced by washing the ore or sand should be submitted to the action of concentrated aqua regia, (consisting of from three to four parts of muriatic acid, to one of nitric,) by which all the gold is extracted. All the platinum, if that metal be present, will be precipitated as below from this liquid.

The solution should then be filtered off with the greatest care, water being afterward poured on to wash the insoluble parts, and to procure the whole of the dissolved gold. Sal-ammonia is then to be added, and if causing a precipitate, the infusion filtered again. This latter is then evaporated to dryness, and alcohol of 0.84 specific gravity repeatedly added, and after digesting poured off, until no more coloured by the dissolving chloride of gold. Iron vitriol (copperas) in solution, if poured into it, will precipitate the pure gold as a brown powder, which may then be filtered, washed, heated to redness, and weighed.

XIII.

Assay of Silver Coins, or Alloys of Silver and Copper.

As a matter of course, with these alloys it is not necessary to perform the operation, which is done with the ores, of first separating the gangue-rock. We proceed with the cupellation, as soon as by prior tests we have ascertained the probable amount of silver and copper. To know this more accurately no method exists, except a previous hasty cupellation, though to a practised assayer it is cognisable from the greater specific gravity, whiter colour, and increased malleability of the more argenteriferous alloys. It is necessary previously to become acquainted with this, as the quantity of lead must be taken accordingly. By adding too much, a loss of silver is

incurred, while, if too little is used, we will not be able to procure a pure silver button, as not all the copper will pass over into the litharge.

The table to regulate the requisite quantity of lead, was calculated by Erker. Still later, D'Arcet arranged another, which, though less simple, experience has shown to be no more accurate, and it is for this reason that I have subjoined the former.

| 32 parts of the Alloy containing | | Require in parts of Lead. | Relative quanti- ties of Copper to Lead. |
|----------------------------------|------------------|---------------------------------|--|
| Parts in Silver. | Parts in Copper. | | |
| 31 | 1 | 128 | 1 : 128 |
| 30 | 2 | 192 | 1 : 96 |
| 28 | 4 | 256 | 1 : 64 |
| 24-26 | 8-6 | 320 | 1 : 40-53 |
| 18-24 | 14-8 | 448 | 1 : 32-54 |
| 8-16 | 24-16 | 480 | 1 : 20-30 |
| 2-8. | 30-24 | 512 | 1 : 16-21 |

From this table, it is evident that the relative amount of lead should decrease as the copper increases, although the more copper the alloy contains, the more lead should be used.

The only way to pulverize a coin or alloy is to file off small portions. About a gramme or fifteen grains of the filings should be carefully weighed off, wrapped in paper, (satin paper is the best, giving little ashes,) to prevent small particles from being lost, and placed in the muffle furnace on the cupel, fig. 2. When the paper is burnt to ashes, the lead is added according to the table. The rest of the assay is exactly as with silver ores. As long as lead and copper are being oxidized, no severe heat should be employed, which is, however, done as soon as the bright flash appears.

The alloy or coin may also be treated according to Gay-Lussac's process, already described. Having thus ascertained the amount of silver, we are easily enabled to calculate that of the copper, by subtraction.

XIV.

*Assay of Gold Coins, or Alloys containing
Gold, Silver, and Copper.*

BEFORE making the regular assay, it is necessary to ascertain the probable contents of the alloy, as with silver. The method commonly used is the *touchstone* or *besanite* test, the same employed by goldsmiths when purchasing coin or bullion. A dark fine-grained basalt or siliceous slate is required, and on this a line is drawn with the gold coin. Those whom business has frequently brought in contact with such alloys are generally able to judge pretty accurately by this alone, as the purer it is, the brighter the yellow, silver making it whitish, copper of a redder hue. To carry this test out farther, the mark is moistened with an acid, which, dissolving the **baser**

metals, leaves the gold in its virgin state.
This acid consists, in thousand parts, of

| | |
|-------|---|
| 784 | parts of pure nitric acid, of 1.340 specific gravity. |
| 16 | “ “ “ muriatic acid, of 1.173 spec. grav. |
| 200 | “ “ “ distilled water. |
| <hr/> | |
| 1000 | |

As with gold ores, several assays should be made at once, to regulate one another. When some of the alloy has been filed off—too small pieces ought not to be taken, on account of their liability to be blown away—these particles should be carefully brushed, to remove the fine dust, which might otherwise only drop off after weighing, and thus cause a decided loss.

For the assays, 5 grains are used in each, and the value of the gold is afterward given in carats fine, pure gold being reckoned at 24 carats fine. In this way an alloy containing 91.666 per cent. of gold will be 22 carats fine, or in other words, it contains $\frac{22}{24}$ or $\frac{11}{12}$ of pure gold to $\frac{1}{12}$ of the alloyed metal.

After having weighed off the above quantity of the gold to be tested, three times the weight of the expected amount of gold, as ascertained by the prior investigations, are taken in pure silver. Some deem two and a half sufficient. The gold and the silver are then carefully wrapped in a piece of paper.

It is now necessary to ascertain the required quantity of pure lead, which varies according to the per-centage of copper in the coin or alloy. As this metal has a much greater affinity for gold than for silver, it is much more difficult to separate it from its alloys with the former than with the latter, and for this reason the lead used should be about twice as much as would be necessary, were we assaying a coin consisting of silver and copper. The following table is given by D'Arcet to regulate the amount to be used :—

| If the contents of gold in the alloy is | | The quantity of lead required is | Relative quantity of lead to the copper. |
|---|-----------------|----------------------------------|--|
| In 1,000 parts. | In carats fine. | | |
| 1.000 | 24. | 1 | 0 |
| 0.900 | 21.6 | 10 | 100.000 to 1 |
| 0.800 | 19.2 | 16 | 80.000 to 1 |
| 0.700 | 16.8 | 22 | 73.333 to 1 |
| 0.600 | 14.4 | 24 | 60.000 to 1 |
| 0.500 | 12.0 | 26 | 52.600 to 1 |
| 0.400 | 9.6 | 34 | 56.666 to 1 |
| 0.300 | 7.2 | 34 | 48.571 to 1 |
| 0.200 | 4.8 | 34 | 42.500 to 1 |
| 0.100 | 2.4 | 34 | 37.777 to 1 |

As with silver assays, if too much be employed, it will produce a loss, while too little would not extract all the copper.

The lead is first placed in the cupel, (fig. 2,) and only when the process of oxidation has commenced, is the paper containing the gold and silver to be added. The rest is performed exactly as with gold ores. The button should be hammered to a very thin sheet, before being submitted to the nitric acid; and to do this well, it should be perfectly cold, as otherwise marginal

cracks are often produced, which again may be productive of loss.

It may not be quite out of place here to give the mint units and the content, weight, etc., of the coins of various countries.

Mint - Units.

| Country. | Mint-unit. | Weight in grammes. | | Finess of principal coins. |
|-----------------------------------|-------------------------|--------------------|--------------|----------------------------|
| | | Fine gold. | Fine silver. | |
| Austria..... { | Gold gulden..... | 6.7258 | — | 900 |
| | Silver gulden..... | — | 11.11 | 900 |
| Brazil..... | Gold milreis..... | 8.8217 | — | 900 |
| Chili..... { | Peso (gold)..... | 1.3725 | — | 900 |
| | Peso (silver). | — | 22.5 | 900 |
| Denmark, Sweden and Norway..... | Krone | 0.4032 | — | 900 |
| England..... | Pound or sovereign..... | 7.3224 | — | 916.66 |
| France, Belgium, Switzerland..... | Franc..... | 0.29 | 4.5 | 900 |
| German Empire... | 10 marks | 0.3584 | — | 900 |
| Greece | Drachme..... | 0.29 | 4.5 | 900 |
| Italy | Lira | 0.29 | 4.5 | 900 |
| Roumania | Lei | 0.29 | 4.5 | 900 |
| Russia | Gold rouble..... | 1.1997 | — | 916.66 |
| | Silver rouble..... | — | 17.9961 | 868.05 |
| Servia..... | Dinar | 0.29 | 4.5 | 900 |
| Turkey | Gold piastre | 0.066 | — | 900 |
| | Silver piastre..... | — | 0.998 | 900 |
| United States of North America. | Dollar | 1.5046 | — | 900 |

The following tables show the content, weight, etc., of the coins of various countries.

Gold Coins.

| Country. | Coins. | Weight in gram'es. | Fineness in thou- sandths. | Fineness in gram'es. | Remedy in weight. | Remedy in fineness. |
|--|---------------------------|--------------------------|----------------------------------|----------------------------|-------------------------|---------------------------|
| Austria... | 8 Gulden .. | 6.4516 | 900 | 5.8065 | 0.002 | 0.002 |
| | 4 " " | 3.2258 | 900 | 2.9032 | 0.002 | 0.002 |
| | Dukaten... | 3.4909 | 986.111 | 3.4424 | 0.00125 | 0.00175 |
| France.. | 100 Francs | 32.2518 | 900 | 29.0323 | 0.001 | 0.002 |
| | 50 " " | 16.1290 | 900 | 14.5161 | 0.001 | — |
| | 20 " " | 6.4516 | 900 | 5.8065 | 0.002 | — |
| | 10 " " | 3.2258 | 900 | 2.9032 | 0.002 | — |
| | 5 " " | 1.6129 | 900 | 1.4516 | 0.003 | — |
| German Empire | 20 Marks.. | 7.965 | 900 | — | 0.002 | — |
| | 10 " " | 3.9825 | 900 | — | 0.002 | — |
| | 5 " " | 1.9912 | 900 | — | 0.004 | — |
| Great Britain | 5 Sovereigns | 39.9403 | 916.667 | 36.6119 | 0.002 | 0.002 |
| | 2 " " | 15.9761 | 916.667 | 14.6448 | 0.002 | — |
| | 1 Sovereign | 7.9881 | 916.667 | 7.3224 | 0.002 | — |
| | $\frac{1}{2}$ " " | 3.9940 | 916.667 | 3.6612 | 0.002 | — |
| Russia ... | $\frac{1}{2}$ Imperial | 6.5440 | 916.667 | 5.9987 | 0.00679 | — |
| | Imperial Dukat... | 3.9264 | 916.667 | 3.5992 | 0.00566 | — |
| | Double eagle..... | 33.4363 | 900 | 30.0926 | 0.000969 | — |
| United States of North America. | Eagle | 16.7181 | 900 | 15.0463 | 0.001938 | — |
| | $\frac{1}{2}$ eagle | 8.3591 | 900 | 7.5232 | 0.001938 | — |
| | $\frac{1}{4}$ eagle | 4.1795 | 900 | 3.7616 | 0.003876 | — |
| | 1 Dollar... | 1.6718 | 900 | 1.5046 | 0.00969 | — |

Silver Coins.

| Country. | Coins. | Weight in gram'es. | Fineness in thou- sandths. | Fineness in gram'es. | Remedy in weight. | Remedy in fineness. |
|--|-----------------------------|--------------------------|----------------------------------|----------------------------|-------------------------|---------------------------|
| Austria... | 2 Gulden.. | 24.6914 | 900 | 22.222 | 0.003 | 0.003 |
| | 1 " " | 12.3457 | 900 | 11.111 | 0.004 | — |
| | $\frac{1}{2}$ " " | 5.3419 | 520 | 2.7778 | 0.010 | — |
| | 20 Kreuzer | 2.6667 | 500 | 1.3333 | — | — |
| | 10 " " | 1.6667 | 400 | 0.6667 | — | — |
| France... | Maria-Theresa- Thaler... | 20.0668 | 833.333 | — | — | — |
| | 5 Francs... | 25 | 900 | 22.5 | 0.003 | 0.002 |
| | 2 " " | 10 | 835 | 8.35 | 0.005 | 0.003 |
| | 1 " " | 5 | 835 | 4.175 | 0.005 | 0.003 |
| | 50 Centimes | 2.5 | 835 | 2.0875 | 0.007 | 0.003 |
| German Empire | 20 " " | 1 | 835 | 0.835 | 0.010 | 0.003 |
| | 5 Marks... | 27.7778 | 900 | 25 | 0.010 | 0.003 |
| | 2 " " | 11.1111 | 900 | 10 | 0.010 | 0.003 |
| | 1 " " | 5.5555 | 900 | 5 | 0.010 | 0.003 |
| | 50 Pfennig | 2.7778 | 900 | 2.5 | 0.010 | 0.003 |
| Great Britain | 20 " " | 1.1111 | 900 | 1 | — | 0.003 |
| | 1 Crown... | 28.2759 | 925 | 26.1552 | 0.004 | — |
| | $\frac{1}{2}$ " " | 14.1380 | 925 | 13.0776 | 0.004 | — |
| | 1 Shilling. | 5.6552 | 925 | 5.2310 | 0.004 | — |
| | 6 Pence.... | 2.8276 | 925 | 2.6155 | 0.004 | — |
| Russia ... | 4 " " | 1.8851 | 925 | 1.7437 | 0.004 | — |
| | 1 Rouble... | 20.7315 | 868.056 | 17.9961 | 0.00857 | — |
| | $\frac{1}{2}$ " " | 10.3658 | 868.056 | 8.9981 | 0.01286 | — |
| | $\frac{1}{4}$ " " | 5.1829 | 868.056 | 4.4990 | 0.01715 | — |
| | 20 Kopecks | 3.5992 | 500 | 1.7996 | 0.01715 | — |
| United States of North America. | 10 " " | 1.7996 | 500 | 0.8998 | — | — |
| | 5 " " | 0.8998 | 500 | 0.4499 | — | — |
| | 1 Dollar... | 27.2156 | 900 | 24.4940 | 0.00357 | 0.003 |
| | $\frac{1}{2}$ " " | 12.5 | 900 | 11.25 | 0.06714 | — |
| | $\frac{1}{4}$ " " | 6.25 | 900 | 5.625 | 0.01428 | — |
| | $\frac{1}{8}$ " " | 5. | 900 | 4.5 | 0.01428 | — |
| | 1 Dime..... | 2.5 | 900 | 2.25 | 0.03571 | — |

Base Coins.

| Country. | Coins. | Weight in gram'es. | Composition. Per cent. | Remedy in content. | Remedy in weight |
|---|-----------------|--------------------------|---|---------------------------------|---------------------------|
| Austria.. | 4 Kreuzer | 13 $\frac{1}{3}$ | Cu | | |
| | 1 " | 3 $\frac{1}{3}$ | | | |
| | $\frac{1}{2}$ " | 1 $\frac{2}{3}$ | | | |
| France.. | 10 Centimes | 10 | 95 Cu + 4 Sn + 1 Zn | 0.01 Cu 0.005 Sn 0.005 Zn | 0.01 0.01 0.015 |
| | 5 " | 5 | | | |
| | 2 " | 2 | | | |
| | 1 " | 1 | | | |
| German Empire | 10 Pfennig | 4 | 75 Cu + 25. Ni 95 Cu + 4 Sn + 1 Zn | 0.005 Ni | 0.05 — 0.05 0.05 |
| | 5 " | 2.5 | | | |
| | 2 " | 3.13 | | | |
| | 1 " | 2 | | | |
| Great Britain | 1 Penny | 9.4498 | 95 Cu + 4 Sn + 1 Zn | | |
| | $\frac{1}{2}$ " | 5.6699 | | | |
| | 1 Farthing | 2.8349 | | | |
| Russia... | 5 Kopecks | 16.352 | bronze | | |
| | 3 " | 9.820 | | | |
| | 2 " | 6.532 | | | |
| | 1 " | 3.244 | | | |
| | $\frac{1}{2}$ " | 1.600 | | | |
| United States of North America | $\frac{1}{4}$ " | 0.800 | 75 Cu + 25 Ni 95 Cu + 5 Sn and Zn | 0.025 | 0.039 0.066 0.044 |
| | 5 Cents | 5 | | | |
| | 3 " | 1.944 | | | |
| | 1 " | 3.944 | | | |

XV.

*To find the Proportion of Gold in a mixture of Gold and Quartz by Calculation.**

THE specific gravity of gold = 19.000

The specific gravity of quartz = 2.600

These numbers can be corrected when experiment shows the specific gravities to be different.

A. Ascertain the specific gravity of the mixture of gold and quartz. Suppose it to be 8.067.

B. Deduct the specific gravity of the

* This article is taken from J. A. Phillips's "Gold Mining and Assaying," (London, 1852,) p. 85, a work published since the first edition of this little volume, and one which, like the other productions of its author, it is needless more particularly to recommend to those who pay attention to the recent publications in this department of applied chemistry.

mixture from the specific gravity of the gold: the difference is the ratio of the quartz by volume:—

$$19.000 - 8.067 = 10.933$$

C. Deduct the specific gravity of the quartz from the specific gravity of the mixture: the difference is the ratio of the gold by volume:—

$$8.067 - 2.600 = 5.467$$

D. Add these ratios together, and proceed by the rule of proportion. The product is the per-centage of gold by bulk:—

$$10.933 + 5.467 = 16.400$$

$$16.4 \text{ is to } 5.467 \text{ as } 100 \text{ is to } 33.35$$

E. Multiply the per-centage of gold by bulk, by its specific gravity. The product is the ratio of gold in the mixture by weight:—

$$33.35 \times 19.00 = 633.65$$

F. Multiply the per-centage of quartz by bulk, by its specific gravity. The product

is the ratio of the quartz in the mixture by weight:—

$$66.65 \times 2.60 = 173.29$$

G. To find the per-centage of gold, add these ratios together, and proceed by the rule of proportion:—

$$633.65 + 173.29 = 806.94$$

806.94 is to 633.65 as 100 is to 78.53

Hence, a mixture of quartz and gold, having the specific gravity of 8.067, contains 78.53 per cent. of gold by **weight**.

XVI.

Assay of Platinum Ores.

THIS metal has as yet occurred in the United States only in gold ores, and even then in the merest traces, and hence, perhaps, it would barely deserve a mention in these pages; but the great interest attached to its occurrence, as so rare a metal all over the world, and its useful application to chemical purposes, has caused me to insert some rules for its assay.

If the ore contain platinum in no larger amount than three or four per cent. of the gold, the former, as already observed in Article VII., on quartation, will be entirely dissolved in the nitric acid used on account of the silver. From this solution of the two metals precipitate the silver with common salt, or muriatic acid, as chloride

of silver; filter and wash until the water dropping from the funnel no longer contains any of the platinum solution. This latter evaporate to dryness, after adding sal-ammonia. Wash it with alcohol, (see Gold, art. XII.) and heat the double chloride of platinum and ammonium to redness, thus producing a spongy mass of pure platinum.

If there be more than three or four per cent. of platinum in the gold, its presence is readily perceived, from various circumstances: thus, in evaporating the lead a higher temperature is necessary than is commonly the case, to make the metal flow and acquire a round form; secondly, the bright light cannot be observed; thirdly, the surface of the button is crystalline or rough, and when large, flat and quite irregular, besides looking dull and having a more or less grayish colour; fourthly, the nitric acid is frequently discoloured; and fifthly, the little roll of gold is not of a

pure gold yellow, but rather inclining towards steel gray.

After having thus recognised a larger quantity of this metal, it becomes necessary, since copper is frequently present, to make a prior test, to ascertain, by cupellation, the amount of the alloy of gold and platinum. After that, two assays should be made; the one, to ascertain the exact conjoint per-centage of the two; the other, inquartation (pure) silver being added, to discover the amount of the gold alone. The difference of the two results gives the amount of platinum. The inquartation silver should not be more than from two and a half to three times the weight of the alloy of gold and platinum; and it is often well to add a certain, accurately weighed quantity of pure gold at the same time, so that the gold may afterward be procured in one connected sheet or piece. This ought particularly to be done, when there is as much as a third the weight of the

gold in platinum, as, for instance, in the platinum grains of the Ural Mountains, which contain about 80 per cent. The button is hammered flat, and proceeded with exactly as gold ores, the platinum dissolving with the silver in nitric acid. But as it does not do so as easily as the latter, at least when in large quantities, it is necessary to repeat the process from the quartering on once or twice, using silver and lead over again. This should be done until nothing but the silver used is dissolved in the nitric acid, or, in other words, until two assays following one another have produced the same results, a thing that may not occur until the fifth time.

It should be remarked, that it is necessary to add a little more lead for cupellation, than would be done if no platinum were in the ore; and also, that just before the bright flash of light occurs during the operation of quartering, it is well to shake the cupel a little, to make the button

stiffen, as soon as the last lead has entered the former, by which the platinum will be more regularly distributed in the alloy, and cannot so well collect in different unconnected lumps, unexposed afterward to the action of the acid. The platinum may then be extracted and reduced from the solution as above.

Electrolytic assay of platinum. Compounds of platinum are decomposed with the greatest ease by the galvanic current, with the deposition of the metal on the negative electrode. A current of two Bunsen cells produces decomposition so rapidly that the platinum separates as platinum black and cannot be determined. If one cell is used it separates in so dense a form that it cannot be distinguished from hammered platinum. It is possible, in this way, gradually to deposit considerable quantities of platinum on the negative electrode without changing its appearance. For the determination of platinum in its salts, the

solution may be slightly acidified with hydrochloric acid or with sulphuric acid, or treated with ammonium or potassium oxalate, gently warmed and electrolyzed. The platinum separates in a comparatively short time. For example, a solution of platinum chloride diluted to 200 cubic centimetres, containing 0.6 gramme platinum, deposited 0.5 gramme in five hours.

Iridium is not reduced from its solutions by a single Bunsen cell. This fact may be used for the separation of platinum from iridium.

XVII.

Assay of Copper Ores—German and Hungarian Method.

THIS ore, if a sulphuret, as is very generally the case, should, after having been reduced to the finest possible powder, be submitted to the process of roasting, vulgarly termed calcining. For this purpose one part by weight of the ore is mixed up with one fifth of graphite, (black-lead,) which, consisting of carbon in a more condensed state than that element occurs in charcoal, is, therefore, so much the more effectual in driving off the sulphur. This mixture of the two should be exposed to an intense red-heat in the cupel, (fig. 1,) (painted over on the inside with red chalk, or Spanish red, to prevent adhesion,) for about twenty minutes, after which it is

to be taken out, and stirred up with a small iron ladle to expose the unburnt parts of graphite, when it should again be exposed in the muffle. In about a quarter of an hour we take it out again, pound it over, for the mass is generally clogged, and mix it with about twice its weight of charcoal-dust, after which we continue the roasting for about one-half to a full hour, according as the ore contains a little or much sulphur, vapours of which may be seen rising during the whole of this process.

After this the ore has a reddish, or what is generally called a ferruginous colour, and we now take it from the first crucible, and introduce it into that represented in fig. 4, Plate II., or if not in possession of such, into a Hessian crucible, fig. 5, on the same plate. For this purpose, however, some *black flux* should be provided. It consists of carbonate of potash and lime, and is made by igniting together one part by weight of saltpetre, and two of com-

mon tartar. The flux produced should be kept carefully corked to prevent the absorption of hygroscopic water. It is still better to make it only when required for immediate use.

If the ore is poor, one tenth part by weight of oxide of antimony, (antimonious acid,) or of arsenic, (arsenious acid,) or if it is richer, fifteen per-cent. of pure lead* are requisite, as will hereafter be seen to make the particles of copper unite. One of these together with three parts of black flux, one half the weight in borax, and two parts of table salt, must then be added to the roasted ore, though none except a part of the

*Some assayers use neither of these three, on the ground that the arsenic, as occasionally even 40 per-cent. may be taken, is very difficult to separate from the copper, and that the antimony may unite with part of the same, forming an antimoniate. They therefore only employ borax and black flux, in about the same proportions, however, as given above. It is hard to say which is best, and it must be left to the discretion and experience of the assayer to act as may be most suitable to his peculiar ores

flux are mixed with the mineral now investigating. The salt is merely used to form a crust over the whole. When all have thus been placed in the crucible, they are covered over with a piece of charcoal, cut to match the size of the vessel; after which the cover is put on. We then expose it to a white-heat for about one-half to one full hour, as may be most convenient, either in the draught furnace, fig. 6, Plate I., or in the muffle furnace; in which latter case, we must allow the longest period of time.

The carbon of the flux is intended to reduce the peroxide of copper produced by roasting, while its carbonate of potash unites with the earthy contents of the ore and the oxides of other metals present, such as iron, which would otherwise also be reduced to their metallic state. With these it forms a slag, the borax being added to make it flow easy, and allow the copper to collect in one button.

On cooling, we break open the crucible, and, on removing the slag, extract a spherical piece of impure or alloyed copper, according as other metals may chance to occur in the ore. If any intermediate crust should have formed between the button and the slag, the ore was not properly roasted, a part of the copper not reduced, and consequently the assay is worthless.

In a good assay the slag should be black and vitreous in appearance, never of an earthy texture. If striated or speckled with red, we may know that protoxide of copper is dissolved in it, and again that the whole cannot be productive of an accurate result. Much attention is required during this test, and, as already mentioned under the heads of silver and gold, the final result depends entirely upon the care taken by the assayer. If well managed, however, as here directed, he can hardly fail to be successful.

The button, as remarked above, may

vary in purity. It generally contains some iron, and (since these metals frequently exist in copper ores) lead, bismuth, tin, cobalt, nickel, antimony, and arsenic—the latter two in particular, if they were added in the process of reduction. Thus it very rarely, if ever, happens, that a copper ore is sufficiently pure to require no third process. The less admixtures the alloy contains, the less brittle and the more ductile it is. Nickel particularly tends to harden it.

To remove the foreign metals, the button is put in a piece of paper, with sufficient borax to cover it, (one-fourth to one-third part by weight, rather more than less,) and if no lead occur in the ore, with about from five to ten per-cent. of that metal, which amount, however, should increase up to forty, or even more, if there are many impurities in the alloy. Even if this be not the case, it is always safer, and can never produce any bad effect, to add much.

A crucible of the kind illustrated in fig. 3,

Plate II., should be brought to a bright white-heat in the muffle. Coals may be placed round to increase the temperature, which should be so great, that the copper, on being introduced, wrapped in paper with borax and lead, may melt in a few minutes.

As long as the tongs held over the button are reflected, or rainbow colours are yet seen to flicker over its surface, lead is still present. As with silver and gold, too great heat ought not to be employed. When the lead has left, we immediately take out the crucible, and immerse it in water, to prevent any copper from oxidating unnecessarily. The button of pure copper is then broken out and weighed. A slight loss cannot be prevented, as it is impossible to prevent some oxide of copper from being formed, and we must therefore grant a larger percentage than the assay would direct.

At least two assays should be made at once, to compare the results, and take the average.

XVIII.

Assay of Copper Ores—English Method.

At the copper mines in Cornwall, a mode of assaying is employed, which in several of its minutiae differs considerably from the one just described. It should be remarked that this method is, properly speaking, only applicable to sulphurets, as all copper contained in the ore as a pure oxide, or combined with an acid, will pass over into the slag during the reduction process; and that therefore if these latter are present, some means should be employed, as shall be shown hereafter, to extract them from the slags, when the other parts of the assay are concluded.

The ore, being a sulphuret, should be roasted as directed in the foregoing German or Hungarian method, after which it

is mixed with from one to one and a half parts of pounded glass, which should, however, contain no lead or arsenic, from twenty-five to fifty per-cent. of saltpetre, and fifty per-cent. of borax. Together with these, it is exposed to a strong melting heat in a clay crucible. On cooling, the button is removed, as is the case in the other mode of procedure, more common on the continent of Europe.

Having thus reduced the copper, it is necessary to purify it, as was also done in the other assay. I must observe, however, that this part of the Cornwall process, again, can only be applied where very little lead is contained in the copper ore.

For this purpose, some *white flux* should be prepared, which is done by igniting together equal parts of saltpetre and tartar. Being as susceptible to the effects of atmospheric moisture as black flux, it requires the same precaution as regards its preservation.

The button of copper alloy is then hammered out flat, to a sheet as thin as it will allow of, without breaking. It is then exposed in a crucible, (fig. 3,) already red-hot, and, as soon as it melts, covered with white flux. Some table-salt, from which the water of crystallization has been removed by heat, is frequently added to give a covering and protect the copper from too immediate contact with the atmosphere, which, together with the heat, would unavoidably create a free oxidation. A considerable ebullition is produced, on the subsiding of which, and when therefore the mass flows quietly, the contents of the crucible are poured into an iron mould, (fig. 6, Plate II.,) greased over beforehand, from which the mass should be removed with a pair of tongs, as soon as sufficiently consolidated, to be dipped in water, which enables us to separate the slag from the copper with greater ease. The purity of the latter is known by its malleability, and by its

not cracking much at the margin, when stretched on the anvil under the blows of a hammer. Should this test prove its yet uncleansed state, it ought again to be submitted to the operation just concluded. Sometimes it is necessary to repeat this several times.

It has already been mentioned, that, particularly when not all the copper in an ore is contained in the shape of a sulphuret, it is impossible to prevent some of this metal from escaping into the slag; and therefore, to diminish this loss as much as possible, English assayers collect the slags, both of the reducing and of the purifying processes, and, pounding them up together, mix them with an equal quantity of tartar and some powdered coke.

This mixture is then melted in a Hessian crucible, after being covered over with common salt. The little button produced in this way is of course impure, and requires the same treatment as the larger one, origi-

nally procured. This second button is weighed together with the large one, and the result will give very accurately the per-centage of copper in the ore.

XIX.

Wet Assay of Copper.

ON account of its greater accuracy and simpler execution, the wet method has, as a general rule, been substituted for the dry assay of copper.

Modified Swedish assay. The cupriferous substance is brought into solution with sulphuric or hydrochloric acid (nitric acid must not be used, as the precipitated copper is again dissolved in it), and the copper precipitated with iron or zinc, and determined either as metal or oxide. This plan is not admissible in the presence of metals which are also precipitated by iron and zinc.

Such metals, however, can be removed during the operation without injurious effect by igniting the precipitated copper, or by a preparatory roasting at not too high a tem-

perature of the assay-sample, first by itself, and then with the addition of some iron pyrites. Tin and antimony are removed by heating with moderately diluted sulphuric acid, then adding nitric acid, and heating nearly to boiling. If necessary, as in the case of metallic sulphides, a few drops of fuming nitric acid are added. The solution is then evaporated to dryness until the fuming ceases. The dry mass is dissolved in hot water and filtered, and the solution treated with some hydrochloric or nitric acid, etc.; *or*, the antimony is removed by fusing the assay-sample with potassium hydrate or potassium carbonate in a silver crucible, lixiviating the potassium antimoniate and dissolving the residue; *or*, the assay-sample is dissolved, neutralized with soda, and digested with a solution of sodium sulphide to extract the antimony, arsenic and tin in soluble form. It is now filtered, and the residue washed and dissolved as above. Ores containing *bitumen*, for instance, cupri-

ferous schists, must be ignited to remove the bitumen, before they are dissolved.

a. *Precipitation with iron.* Into a suitable flask placed in an oblique position bring, according to the richness of the ore, 1 to 5, generally 2.5 to 5, grammes of the assay-sample, and decompose them by heating with sulphuric acid, adding from time to time some fuming nitric acid or potassium chlorate, until the separated sulphur, inclosing particles of the ore, is oxidized as much as possible. *Or*, the sample is at once dissolved in aqua regia. It is then evaporated to dryness with some sulphuric acid, or until the sulphuric acid vapors appear in the flask. To dissolve the basic salts, add a few drops of sulphuric acid to the dry mass and then, cautiously, water. The fluid, which is now entirely free from nitric acid, is filtered into a flask, and the residue washed until the wash-water no longer produces a red stain upon a piece of bright sheet-iron. Two pieces of iron wire, 3 to

4 centimeters long, are then added; or, to shorten the time required for the assay, the fluid may at once be filtered into a porcelain dish in which the iron wires lie, and copper will then be precipitated during filtration. The filtrate is then sufficiently diluted and gently heated until a pointed iron wire dipped into the fluid does not show a reddish stain of copper. To prevent the separation of basic iron salts, which are more readily formed by hot water, the copper is twice decanted with *cold* water into a spacious beaker and then three times with boiling water. The flask is then completely filled with cold water, and a flat-bottomed porcelain dish about 20 millimeters high and 80 millimeters in diameter is placed bottom upwards, on top of it. The flask and dish are then inverted. The mouth of the flask is held in an oblique position, and the water allowed to run into the dish until it is nearly full. The flask is left standing in the dish until all the

copper and the iron wires have fallen into the water in the dish. Small particles of carbon separated from the iron will remain floating on the water for some time. The flask is then gently removed by drawing it over the side of the dish, which should be somewhat inclined for the purpose. The iron is freed from copper by rubbing with the fingers, which should be rinsed off in the water. The copper is now decanted twice with boiling water. This is poured off as completely as possible from the copper, which is moistened with absolute alcohol, and dried on the water-bath until it has assumed a pulverulent condition. It is allowed to cool in the desiccator, and is then brought upon the pan of the balance, or into a tarred porcelain crucible with the aid of a fine brush, and quickly weighed. It is now dried for 10 or 15 minutes more, and again weighed until the results agree; or, the copper is spread out upon a roasting dish and ignited in the muffle furnace, and

the metal calculated from the amount of cupric oxide formed (100 cupric oxide = 79.88 copper). If the water used in decantation shows a reddish sediment, it should be filtered, the filter dried and ignited upon the scorifier, and the percentage of copper resulting from the cupric oxide should be added to the principal yield.

Correction for iron that may be contained in the precipitated copper on account of a deposit of basic iron salts: The precipitated copper is ignited upon the scorifier until it becomes black. The cupric and ferric oxides formed are weighed and dissolved in hydrochloric or sulphuric acid. The ferric oxide is precipitated with ammonia. The solution is filtered upon a small filter of paper. The filter is dried and ignited, and the ferric oxide, which may be found, is deducted from the combined weight of the cupric and ferric oxides, and the copper calculated from the quantity of pure cupric oxide found. Instead of decanting the pre-

precipitated copper, it may all be filtered, dried, ignited, weighed, and dissolved, as above, for obtaining the content of iron.

Pure precipitated copper has a fine copper color. If the solution contains antimony and arsenic, it has first a copper color which later on, by the antimony and arsenic being precipitated, changes to black. The larger portion of the antimony, after the precipitate has been evaporated to dryness with sulphuric acid and again moistened with water, remains as basic sulphate of antimony, while arsenic passes into solution. The residue from the solution of the ore, etc., is tested for copper by heating it with nitric acid, filtering, and adding ammonia in excess. The appearance of a blue color indicates copper.

Instead of two iron wires, a single wire bent into the form of a ring, with one end projecting vertically, may be used. The ring is dipped into the liquid in the beaker glass so that the end projects. When pre-

cipitation is complete, the copper is rinsed from the ring, decanted, etc., as above. *Or*, a strip of sheet-iron may be used in place of the wire, but it must be immediately removed from the liquid after the precipitation of the copper is complete to avoid the formation of basic iron salts. In solutions that are too concentrated, the copper adheres too strongly to the iron.

b. *Precipitation with zinc free from lead and arsenic.* Prepare a solution of the assay-sample with sulphuric acid, as above described, and filter. Then place a strip of zinc in the solution and heat until a bright iron wire held in it shows no copper deposit; or until a drop of the solution placed upon a porcelain dish is not browned by sulphuretted hydrogen. The strip of zinc is then taken out, and the precipitated copper washed off with the wash bottle. It is next filtered until but a small layer of water covering the copper remains. A few drops of warm hydrochloric acid are then added

to dissolve any particles of zinc which may be present. It is now decanted, etc., as in the precipitation with iron; or it is filtered as soon as effervescence ceases, quickly washed with hot water, and dried. The copper is then detached from the filter, ignited on the cover of a porcelain or platinum crucible, or upon a roasting dish in the muffle and the oxide quickly weighed. The black crust upon the end of the piece of zinc, which has been dipped into the fluid, is a spongy layer of zinc colored by a trace of sulphide of copper. Nickel, which is not thrown down by iron, is precipitated with zinc, but cobalt is not.

Granulated zinc may be used instead of a strip, but the granules must be completely dissolved by the time the bubbles cease. The copper is then decanted, etc. The cuprififerous fluid may be filtered into a platinum dish, and some hydrochloric acid added to it. Now heat and add a few small pieces of zinc, whereupon the copper will

firmly deposit itself on the platinum, but loosely on the zinc. After precipitation is complete, which is ascertained by testing with sulphuretted hydrogen, as above, the copper is rubbed and washed off from the zinc. It is then allowed to settle, decanted, treated with hot water, to which some hydrochloric acid has been added; then quickly washed with hot water by decantation to prevent the loss of copper by solution. It is finally moistened with absolute alcohol dried in a water-bath at 230° to 248° F., and the tarred platinum dish, after having been cooled in the desiccator, is weighed. If the utmost accuracy is demanded, the copper is heated in a current of sulphuretted hydrogen.

XX.

Electrolytic Determination of Copper.

CLASSEN'S method for the electrolytic determination of metals is based upon the fact that the separation of the metal is best effected when the oxide is fixed to an acid readily decomposed by the current, so that a secondary reaction cannot take place. Such an acid is oxalic acid, which splits into carbonic acid and hydrogen. Most heavy metals give insoluble precipitates with oxalic acid; their double alkaline salts are, however, readily soluble, and by adding ammonium oxalate in excess the reaction progresses with ease and without the formation of a precipitate. The carbonic acid separated on the positive pole by the decomposition of the ammonium oxalate combines with the ammonium, forming am-

monium carbonate. In general the process is conducted by converting the neutral chlorides and sulphates of the metals into double oxalates by the addition of a large excess of ammonium oxalate, heating the solution and exposing it to the action of a galvanic current, whereby the metals deposit quickly and in a compact form on the negative electrode.

The determination of copper, as is well known, can be very easily effected directly from the acid solution. According to Claisen's method the solution (concentrated, if necessary, by evaporation) is heated to boiling, and, after adding 3 to 4 grammes of solid ammonium oxalate, subjected to the action of the electric current as soon as everything is dissolved. The copper separates quickly and easily, provided the current is not too weak. With a strength of current corresponding to 300 cubic centimeters of oxyhydrogen gas per hour, 0.15 gramme of metallic copper can be separated

in 25 minutes. As the negative electrode, Classen uses a platinum dish, and for the positive electrode a disk, 4 to 5 centimeters

in diameter, of moderately thick platinum sheet, Fig. G, which is secured by a screw to a medium thick platinum wire.

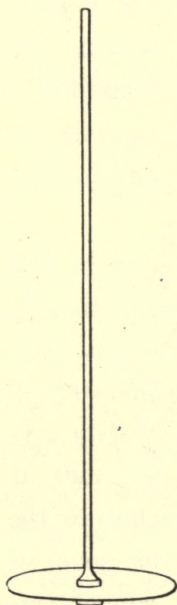


FIG. G.

To prevent loss the platinum dish is covered with a watch crystal perforated in the centre. The entire arrangement of the apparatus is shown in Fig. H. The glass cylinder shown in front is the resistance-arrangement recommended by Classen for the reduction of more considerable strengths of current.

It consists of a rod *b* with a zinc pole, which can be moved to and from the zinc pole *a* until the current reaches the desired strength. The zinc poles must be amal-

gamated with mercury and hydrochloric acid, and the contacts at *a*, *b* kept clean.

According to Herpin's method of assaying copper by electrolysis, 1 gramme or more of the assay-sample is dissolved in nitric acid. The solution is evaporated

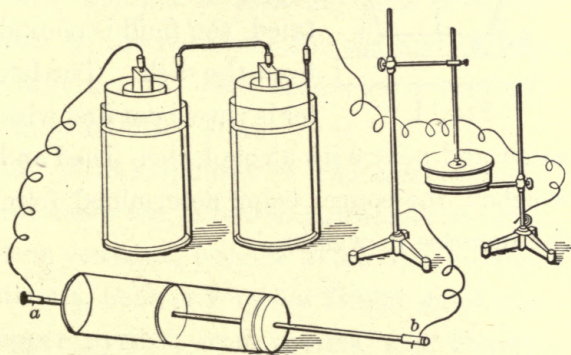


FIG. H.

nearly to dryness, the residue dissolved in a small quantity of dilute sulphuric acid and the solution diluted to 60 or 70 cubic centimeters. The solution is poured into the platinum dish *A*, Fig I, and the conducting stand *B* of the dish is connected with

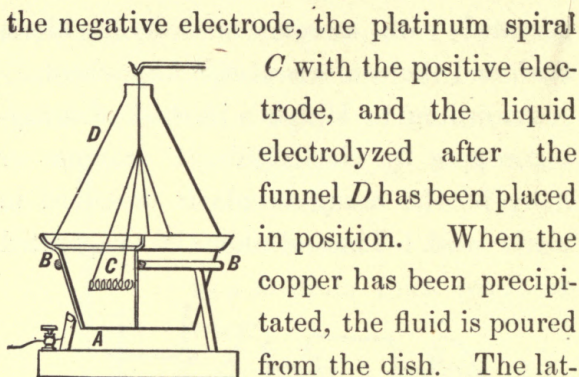


FIG. I.

the negative electrode, the platinum spiral *C* with the positive electrode, and the liquid electrolyzed after the funnel *D* has been placed in position. When the copper has been precipitated, the fluid is poured from the dish. The latter is rinsed out first with water and next with alcohol, then dried and weighed, the copper being determined from the increase in weight.

XXI.

Assay of Lead Ores.

THE method of assaying lead ores depends on the nature of the ore and chiefly on whether the lead occurs in a sulphuretted or oxidized state.

A. With sulphuretted lead combinations three cases are to be distinguished : *a.* The galena contains, besides a few earthy constituents, none or very few foreign sulphides (copper pyrites, iron pyrites, zinc blende, etc.). *b.* Many of such sulphides are present. *c.* Earthy constituents are present in predominant quantity.

As a rule, these conditions can be better recognized, when the assay-sample is in pieces than when in the form of a powder. In the latter case elutriation may, with the assistance of a magnifying glass, give

important disclosures regarding the metallic admixtures.

1. *Methods of assaying sulphuretted lead ores which contain few or no foreign sulphides.*

These methods are based upon the desulphuration of the galena by iron and alkalis, or, as is more generally done, simultaneously by iron and alkalis, and promoting the reduction of the alkalis by the addition of carbon, or entirely omitting the latter. Too much carbon causes refractoriness. The carboniferous substances also maintain an oxidizing atmosphere in the crucible and prevent the oxidation of the sulphides of iron to oxidized iron, which strongly attacks the sides of the crucible and in pouring out the assay contributes to the retention of particles of lead. Any white lead or sulphate of lead occurring together with the galena are reduced by the carboniferous substances. By the alkalis alone, or the simultaneous use of suitable

fluxes, such as glass and borax, the earths occurring in the ores are scorified. If foreign sulphides, such as copper pyrites, sulphide of antimony, zinc blende, etc., are associated with the galena, they are also partially decomposed by the alkalies and the iron, and their radicals pass into the lead. They also give rise to the formation of sulphur-salts, which retain sulphide of lead in the slag. Of the alkaline combinations potash and dehydrated soda are chiefly used. The carbon is employed either in the form of coal dust mixed with the potash, or still better, in a finely divided state in black flux, or in a mixture of potash and flour.

The *black flux* is prepared by pouring a mixture of $2\frac{1}{2}$ parts crude tartar and 1 part saltpetre (*crude flux*) in a heated clay crucible and laying a glowing coal upon the surface; or by gradually introducing the mixture into a glowing crucible. The mass thereby decrepitates, whereby the oxygen

of the nitric acid first oxidizes the hydrogen of the tartaric acid, this being the most readily oxidizable constituent, and then also a portion of the carbon, while, with a correct quantity of saltpetre, another portion of the carbon separates in a finely divided state. By taking too much saltpetre all the carbon is oxidized, and instead of a mixture of potassium carbonate and carbon (*black flux*) only potassium carbonate (*white flux*) is obtained, and with little carbon, *gray flux*. Black flux has the disagreeable property of readily absorbing moisture, and, hence, must frequently be prepared fresh. For this reason it is more convenient to use a mixture of 100 parts potash and 10 to 15 parts rye or wheat flour. On heating the mixture the latter carbonizes and finely divided carbon is separated with the evolution of reducing varieties of gas.

The fluxes are frequently protected by a cover of common salt. It serves to keep off the air from the melting assay and after

it has become thinly-fluid as a wash, bringing down from the sides of the crucible adhering metallic grains or fluxes.

All assays, containing in their charge carbon, puff up in consequence of the formation of carbonic oxide, and hence capacious crucibles, which should at the utmost be filled $\frac{2}{3}$ full with the assay-sample, have to be used. The crucibles are heated either in an air-furnace or a muffle-furnace; a blast-furnace being seldom used.

For assaying lead ores of the kind mentioned under A 1, various methods are in use in different places.

a. *Assays without fluxes in iron vessels.* These are suitable only for nearly pure galena.

a. *Assay in iron dishes.* Galenas nearly free from foreign admixtures are assayed in England as follows: About 300 grammes of the ore are placed in a red-hot wrought-iron dish. The latter is about 5 to 6 millimeters thick, 0.127 meter in diameter and

0.025 meter deep in the centre. After introducing the assay-sample the dish is provided with a semicircular lid of somewhat larger diameter, and the whole heated in a blast-furnace for about 2 minutes. The dish is then taken from the furnace and placed upon a ring. When the matte is congealed, empty the still liquid lead into a conical bronze mould, melt the matte in the dish once more, using a stronger fire, and then pour all upon a metal plate, whereby a small lead regulus still results. In 5 to 6 minutes two such meltings are finished. Seventy-five to eighty per cent. of lead are in a short time brought out, and these assays are especially used for controlling the reverberatory furnace process, in which nearly the same volatilization of metal takes place.

β. *Assay in iron crucibles.* In South Wales about 80 grammes of galena are melted in an iron crucible, about 72 millimeters high and 69 millimeters in diameter

on the top, in an air-furnace for about 10 to 15 minutes. The yield is 81 to 82 per cent.

b. *Assays with fluxes.* The fluxes, which have to be added to more impure ores, serve either for decomposing the galena or for scorifying oxide and earthy admixtures, or for both purposes.

Of silver nearly everything passes into the lead; with a content of sulphide of iron in the slag more silver is retained than without such a content. Copper is divided between lead and slag. Of zinc only a small portion passes into the lead, that which does not volatilize passing chiefly into the slag. Iron is only found in traces in the lead, but antimony in abundance, while arsenic partially volatilizes as arsenic and orpiment, and partially yields speiss with iron. Silica promotes the scorification of lead more than calcareous spar and heavy spar. If lime and ferric oxide be at the same time present

scorification is counteracted. The lead in an impure lead regulus obtained by the dry assay can only be determined by the wet method. With a content of copper, dissolve the regulus in moderately strong nitric acid, evaporate the solution with sulphuric acid until the nitric acid is expelled, dilute with water, bring the sulphate of lead upon a dry, weighed filter, or reduce it with cyanide of potash and soda in a porcelain crucible. Brittle lead containing antimony is oxidized with nitric acid, the dilute solution digested with ammonia, ammonium sulphide and some sulphur, and the undissolved sulphide of lead converted into sulphate.

According to the quality and quantity of the foreign admixtures, the following methods of assaying may be used.

a. Assay in an iron crucible with black flux. With ores as free as possible from copper and poor in earths, this assay in a short time gives the greatest yield ($84\frac{1}{2}$ to

85½ per cent.), because it is for a shorter time (10 to 15 minutes) in the fire and can be executed with larger quantities of ore, whereby the loss is distributed over larger quantities. The absence of a larger content of earths favors the complete pouring out of the contents of the crucible into moulds, while with larger quantities of slag, the latter adheres to the sides of the crucible and mechanically retains lead. This method, which is much used in England, Germany, France and Belgium, is executed as follows: Heat a wrought-iron crucible in an air-furnace to a red heat, and then charge it with 25 to 50 grammes of lead ore and 12 to 15 grammes of flux (potash, soda, or a mixture of 2 parts salt-petre, 3 argol, and 1 borax). Then add a cover of common salt or powdered glass, place an iron lid upon the crucible and gradually heat during 5 minutes to a red heat, and maintain this temperature for 5 to 8 minutes longer. When all is melted,

and any globules of lead appearing on the surface or unmelted masses have been submerged with a spatula, take the crucible from the furnace, and, after allowing it to cool somewhat, pour the contents into a warmed ingot-mould painted inside with black lead. The slag may be again melted with some flux (soda or potash and argol) for 10 to 12 minutes.

The difference between the quantity of lead obtained by the assay and that actually present in the ore is nearly the same with ores from the highest to 50 per cent. content of lead, but increases with poorer ores. Of 86.81 per cent. actual content of galena $84\frac{1}{4}$ to $85\frac{1}{4}$ per cent. is obtained by this assay. The proportions of the charge in various works are as follows: In *England*: for rich ores, 500 grains (32.4 grammes) ore, 500 grains soda and 50 grains argol; for poorer ores, 850 grains soda, 150 grains borax and 50 grains argol; time for melting, 8 to 10 minutes; entire time, 10 to 15

minutes. At *Freiberg*: 50 grammes ore, 2 tablespoonfuls flux (3 parts argol, 2 saltpetre, 1 borax); time for melting at a red heat, 12 to 15 minutes; time for heating the slag with potash, 10 to 12 minutes.

β. *Assay in a clay crucible with black flux and iron; available for ores poor in foreign sulphides but richer in earths.* At *Freiberg* this method of assaying is executed as follows: Charge a crucible, first with 3.75 grammes of powdered ore, then, according to its richness, place upon the ore 0.37 to 1.12 grammes of iron wire (a piece 6.5 to 9.8 millimeters long and 4 to 5.5 millimeters thick), spread out upon it 11.25 grammes of black flux, or 8.37 grammes of a mixture of potash and flour, cover the latter with 1.12 to 1.5 grammes of borax, and finally add a cover of common salt a few millimeters thick. If foreign sulphides, combinations of antimony or arsenic are present, the ore previously to charging it may be somewhat roasted.

With galena ores the charge at Pribram is as follows: 5 grammes ore, 11.5 to 12 grammes black flux (2 parts argol, 1 saltpetre), 1 to 1.2 grammes borax and 2 to 2.5 grammes iron wire. With ores containing blende: 5 grammes ore, 20 grammes black flux, 1 to 1.2 grammes borax and 2 to 2.5 grammes iron wire. Some argol is always placed upon the bottom of the crucible and under the common salt serving as cover, 2 to 3 per cent. saltpetre, in order to make the common salt more fluid and retain the lead, which is volatilized, by oxidation.

The assay is melted in an air-furnace. When taken from the furnace, the crucible is allowed to cool, the lead regulus is then freed from slag, flattened to remove any adhering sulphide of iron, and weighed. The assay may be considered a success when assay and counter-assay agree, when the lead is at the bottom in *one* button and the slag uniform in character. By a few

blows of the hammer, the iron wire is separated from the adhering lead.

The galena is at first desulphurized by the alkali, alkaline sulphate and potassium sulphide being formed besides lead. The potassium sulphide combines with a portion of still undecomposed galena to a sulphur-salt which passing into the slag at an increased temperature—hence, towards the end of melting—is freed by the iron from its content of lead. The carbon prevents an oxidation of the lead and reduces the alkali. It, therefore, also contributes towards the desulphurization, the potassium formed being closely allied to sulphur. Another portion of the alkali in conjunction with the borax and glass causes the scorification of the earths and metallic oxides contained in the assay-sample. Besides silicates the slag also contains sulphur-salts, especially a double sulphide of potash and iron. The cover of the readily-fluid common salt prevents the adherence of lead globules on

the edge of the crucible and shuts out the air.

This method of assaying is very simple, readily executed and permits of the best yield of lead for ores of the above-mentioned kinds. From galena with 85.5 per cent. of lead up to 84.85 per cent. is obtained; with more impure ores, the loss of lead may, however, increase to 10 per cent.

γ. Upper Harz potash assay in clay crucibles. Melt 5 grammes of galena with three times the quantity of potash under a cover of common salt in a crucible under the muffle strongly heated for 15 to 30 minutes or longer until fusion is complete. The following reaction thereby takes place:

$$4 \text{ K}_2 \text{ C O}_3 + \text{Pb S} = 4 \text{ Pb} + 3 (\text{K}_2 \text{ S} + \text{Pb S}) + \text{K}_2 \text{ S O}_4 + 4 \text{ C O}_2.$$

In order to separate from the sulphur-salt ($\text{K}_2 \text{ S} + \text{Pb S}$) the lead retained by it open the muffle 5 to 10 minutes and moderate the fire, whereby the $\text{K}_2 \text{ S}$ is converted into $\text{K}_2 \text{ S O}_4$, and the liberated Pb S partially

into Pb S O_4 . If now the muffle is again closed and after 10 minutes a strong heat is given, the sulphate of lead acts upon the decomposed sulphide of lead, metallic lead being formed, if the proper time has been allowed for the above-mentioned cooling ($\text{Pb S} + \text{Pb S O}_4 = 2 \text{ Pb} + 2 \text{ S O}_2$). If cooling is continued for too long a time, oxide of lead remains in the slag, and if for too short a time, sulphide of lead. The duration of cooling at which the most lead is obtained must be ascertained for each ore.

2. *Methods of assaying sulphuretted lead ores which contain many foreign sulphides.*

The above-mentioned assays are not suitable for such lead combinations, because the foreign sulphides are decomposed by the alkalies and the iron, their radical passing into the lead button, and an abundant formation of sulphur-salts takes place, which always retain much lead in the slag.

For such substances the calcination-assay

and a partially wet assay with sulphuric acid are suitable.

a. *Calcination-assay.* This assay is used for Rammelsberg ores whose small content of lead is diminished 5 to 8 per cent. by the presence of considerable quantities of iron pyrites, copper pyrites, zinc blende, antimony and arsenic combinations, as well as earthy substances. Take from a pile of ore, which has previously been roasted, three times, 5 to 10 grammes. Pulverize this assay-sample and thoroughly roast it for 6 or more hours in a more or less shallow crucible about 65 millimeters in diameter, 13 to 16 millimeters deep and painted inside with redde or chalk washes. By thorough roasting is understood heating the ore at a gradually increasing temperature and with the access of air, so that no fusion takes place, but the air can exert an oxidizing effect upon the sulphides, whereby the latter, with the development of sulphurous acid, are converted into oxides.

In order to effect this as completely as possible frequently turn the crucible around so that the air can act uniformly. After half an hour take the crucible from the furnace, triturate the assay-sample, if it should be slagged together, in a cast-iron mortar, roast again for about half an hour, triturate again, mix coal dust or another carbonaceous substance, for instance, suet, with it and reheat. In roasting, metallic sulphates are also formed from the sulphides, which by the addition of carbon are to be decomposed so that, with the development of sulphurous acid, they again form sulphides and then with the repeated access of air are reconverted into oxides and sulphates. The sulphur can only be removed by repeated triturating and heating of the ore with carbon at a gradually increased temperature. The last portions of it, which remain as sulphates in the roasted powder, are removed by heating the latter with ammonium carbonate, volatile ammonium

sulphate being formed. The sulphate of lead formed in roasting the sulphide of lead can in this manner be but incompletely decomposed and hence remains as such in the roasted sample. A thoroughly roasted sample has an earthy appearance and shows no odor of sulphurous acid. It is finally taken from the furnace, allowed to cool and, after being charged with suitable fluxes, melted for metallic lead.

The roasted sample chiefly consists of the oxides of the lead, iron, copper, zinc, etc., and if it is melted at not too high a temperature with reducing agents (black flux, potash and flour) and solving fluxing (borax, glass), the lead oxide is reduced to metallic lead while the metallic oxides, reducible with more difficulty, are scorified. However, with too high a temperature in melting, the latter are also partially reduced and the resulting lead is impure. Too much or too little of solving fluxes causes scorification of lead oxide, or reduction of

the foreign oxides if scorifying agents are wanting. The sulphate of lead present is partially decomposed by the acids of the borax and glass and partially reduced to sulphide of lead, from which by the alkali a portion of the lead is separated, while another portion remains as a double sulphide of potash and lead in the slag. For the decomposition of the latter combination a small addition of iron is recommended.

Melting is executed by mixing the roasted assay-sample with three to four times the quantity of black flux and an equal weight of borax and glass, bringing the mixture into a crucible, covering it with common salt and, after placing the lid upon the crucible, heating it in an air-furnace for about $\frac{1}{4}$ hour. If the assay has been successful the lead lies on the bottom under the well-fused slag in *one* button without being enveloped by brittle sulphides.

b. *Assay with sulphuric acid.* Decom-

pose 5 or 10 grammes of finely pulverized ore in a digesting-glass with nitric acid, evaporate, with the addition of sulphuric acid, the contents of the glass to dryness, moisten the dry mass with a few drops of sulphuric acid, soften it with water, bring it upon a filter, and thoroughly wash the residue upon the latter. Besides insoluble earths, the residue chiefly consists of sulphate of lead which is with difficulty soluble in water, while the sulphates of the metals associated with galena being readily soluble in water are removed after washing the residue. The filter is dried, ignited with its contents and the ignited mass melted with black flux and iron, whereby the sulphide of lead reduced from sulphate of lead is decomposed by the iron, the result being a button of lead.

This assay gives a more correct content of lead than the preceding, and is also suitable for lead alloys. On account of the availability of lower temperatures, the

assay becomes still more accurate by mixing the residue obtained by treating the mass in a capacious porcelain crucible and heating until the free sulphur is volatilized, with about five times the quantity of potassium cyanide (with or without soda) and melting in the covered crucible over a small lamp. The cold melt is extracted with water, and the lead remaining behind in one or more buttons decanted with water containing alcohol, dried and weighed. If a black powder, which, however, contains no sulphide of lead, has been formed, remove it by washing. In both cases a content of antimony passes into the lead and has to be removed by the wet process.

3. *Methods of assaying for sulphuretted lead ores, which contain many earthy constituents.* In such combinations the content of lead can only be very inaccurately determined by the dry method by melting a larger quantity of assay substance (100 to 500 grammes) with the most powerful

fluxes (caustic alkalies), and the addition of reducing agents, and stirring a piece of sheet iron bent in the form of a horseshoe around in the fluid mass until all the lead is separated. If the content of lead is not too small, not below $\frac{1}{2}$ per cent., a useful result is also obtained by mixing 10 to 20 grammes of the substance to be assayed in the ordinary manner with potash or black flux, spreading out on top of the mass an accurately weighed quantity of finely divided silver (30 to 50 per cent.), covering the latter with common salt and melting in the usual manner. The increase in the weight of the silver represents the content of lead.

B. *Oxidized lead ores and smelting products* may be assayed as to their content of lead by two methods, viz. :

1. *By melting with reducing agents* when the assay-sample contains free lead oxide (litharge, lead scum, or skim) or lead oxide fixed on carbonic acid or an organic acid

(as white lead ore; lead acetate), or lead oxide combined with fixed mineral acids which cannot be reduced, but combine directly with the alkalies (silicate, phosphate, tungstate and molybdate of lead; in chromate of lead, the chromic acid is reduced to chromic oxide, which passes into the slag).

Combinations of the above-mentioned variety are mixed with three or four times their weight of black flux or potash and flour, and melted under a cover of salt in a crucible. The slag must become thinly-fluid, and as this chiefly depends on the nature of the by-constituents, the height of the temperature in melting, and the duration of the latter, must accordingly be modified. To highly silicated lead slags it is best to add a flux of from 20 to 50 per cent. borax, and to very poor slags, containing $\frac{1}{4}$ to $\frac{1}{2}$ per cent. of lead, a flux of 15 to 20 per cent. silver for the collection of the lead. Slags containing sulphur are

somewhat roasted, or receive a flux of from 5 to 10 per cent. iron. According to the refractoriness of the slags, melting may last from $1\frac{1}{2}$ to 2 hours, in order to give the lead an opportunity to thoroughly separate from the very thinly-liquid slag. At Mecher-nich the lead slags are melted with $\frac{2}{3}$ soda and $\frac{1}{3}$ borax in an iron crucible at a bright red heat; result, 0.4 to 0.6 per cent. of lead.

Percy recommends the following charges: For white lead ore: 500 grains ore, 500 soda, 100 argol and 30 borax. Time of melting, 20 minutes. For minetite (green lead ore): besides iron flux, 300 grains ore, 400 soda, 20 coal dust and 30 borax; or 350 grains soda, 100 argol and 30 borax. Duration of the process from the time of introducing the charge to pouring out, 25 to 30 minutes.

2. *By melting with reducing agents and iron*, when the lead oxide is fixed on sulphuric acid, arsenious or arsenic acids, as, for instance, in sulphate of lead, in calcined

galena and matt of lead, lead smoke, slag-lead, dross, etc. Charge with three to four times the quantity of black flux and 10 to 30 per cent. iron. For slags, an addition of 5 to 10 per cent. iron suffices.

XXII.

Assay of Lead Ores by Heat.

THERE is no way of assaying lead ores by heat which gives the full amount of the metal, as it is so easily oxidized by a high temperature. Generally, the loss varies from one-sixth to one-twelfth, or yet more commonly is about a tenth; and for this reason, the result of the assay should afterward be increased by $\frac{1}{10}$, on giving the percentage of lead.

The assay is performed in the crucible, fig. 4, or in the Hessian one. The part of ore used ought to be about two hundred grains, as lead ores (galena, a sulphuret of lead is the most common) are very heavy, and therefore are comparatively small in bulk. Three parts, or here six hundred grains, of black flux are mixed up with the

powdered ore.

A little piece of very thick wire, or of a round iron bar, weighing from thirty to forty per-cent. of the weight of the lead ore, is placed on the top, in the crucible.

We then expose the whole to a strong, steady red-heat, for about an hour, in the bellows furnace, fig. 6, Plate I. This is effected by first placing the crucible on the brick, and then making a layer of cold burnt coals, as high as the brick. On this come the live coals, and on them the unburnt ones up to the top of the furnace. When burnt down, we take out the crucibles, and thus obtain a button of pure lead and some slag, besides a remaining portion of the iron.

The latter should be added in excess, to insure the entire absorption of the sulphur. Galena contains, in hundred parts, 13.45 parts of sulphur to 86.55 of lead, which would require 22.67 parts of metallic iron to form sulphuret of iron, consisting of 37.23

parts of sulphur to 62.77 of iron. If, therefore, two hundred grains of the ore are used, the iron should weigh from 60 to 100 grains. Filings ought never to be used, as they are always covered, to a smaller or greater amount, with oxide. Besides, it is very difficult to get them as free from impurities as wire. Weighed pieces of the latter should be kept in some vial or box for this purpose.

XXIII.

Assay of Lead Ores by a Wet Process.

THIS method will be found much the more accurate of the two, although it may not often happen that a practical assayer has sufficient time for this proceeding. (See Woehler's Anal. Chem.)

The ore (galena) should be powdered much finer even than in the assay by heat, after which it is moistened with fuming nitric acid, and digested in the sand-bath, by which process it is entirely changed from the sulphuret to the sulphate of lead.

If the mass be diluted with water and filtered, the merest traces only of lead can be found in the solution,—quantities too small to deserve any farther attention. If the ore contain copper, iron, or silver, they will be contained in this filtered solution;

the first are then discovered by ammonia, the latter by muriatic acid.

Should fuming nitric acid not be at hand, and if therefore a weaker kind is used, a mixture of sulphate of lead and sulphur is produced, together with a solution of nitrate of lead. From this latter the metal should be precipitated with sulphuric acid. By heating the dried residue,—after filtering and washing, by pouring water over it while yet on the filter,—sulphur is evaporated and sulphate of lead is retained.

In both cases, the lead produced is a sulphate, and in this shape it should be weighed, and from the result the amount of the former alone may be easily calculated, as will be seen from the sequel.

Sulphate of lead consists, in 100 parts by weight, of

Oxide of lead . 73.56

Sulphuric acid . 26.44

100.00

and the oxide of lead, again, of

Lead 92.83

Oxygen 7.17

Hence we say, if 100 parts of oxide of lead contain 92.83 of pure lead, then 73.56 of pure lead contain 68.285748 parts; or, in other words, sulphate of lead consists of

Lead 68.285748

Oxygen 5.274252

Sulphuric acid . 26.440000

100.000000

For this assay about twenty grains only need be taken. A smaller amount might make us incapable of finding the real quantity of lead, while a larger one would only give us unnecessary trouble.

XXIV.

Gravimetric Analyses of Lead Ores.

At Bleiberg, in Carinthia, this method is executed for pure galenas as follows: Decompose 2 grammes of the ore with concentrated nitric acid, add a few drops of sulphuric acid, evaporate nearly to dryness, soften the mass in water, wash, digest the contents of the filter (lead sulphates, calcium and barium, sulphur, silica) with concentrated soda solution for one hour in order to convert the sulphate of lead into carbonate, filter, wash the filter until a drop of the filtrate no longer shows turbidity with barium chloride, boil the residue with dilute nitric acid or with acetic acid, wash with water until acid reaction ceases, precipitate the lead with as little sulphuric acid as possible (an excess of acid also pre-

precipitates lime), filter off the sulphate of lead, wash, dry and heat to a red heat, whereby differences exceeding 0.1 per cent. should not occur. In order to avoid with ores poor in lead and rich in lime, bringing from the start too much calcium sulphate amongst the lead sulphate, the evaporation of the nitric acid solution with sulphuric acid is not carried quite so far, and the solution diluted with $\frac{1}{2}$ liter water before adding the sulphuric acid.

Storer decomposes 2 to 3 grammes of galena with a regulus of zinc 6 millimetres in diameter and 7 millimetres thick, and 100 to 150 cubic centimetres of hydrochloric acid diluted with four times the quantity of water, and heated to 122° F. for 15 to 20 minutes. He then decants the metallic lead, separated under the development of sulphuretted hydrogen, with hot water, cleanses the cylinder of zinc by rubbing with the finger and rinsing, and dries the lead in a tared porcelain crucible at a

moderate temperature in a current of illuminating gas, or in the air-bath. In the presence of insoluble gangues the lead is dissolved from them by means of nitric acid and the residue weighed. Other metals precipitable by zinc have to be separately determined by special assays, and deducted from the lead. Since the lead sponge is readily oxidizable, Roessler fixes it on a weighed quantity of Wood's metal (1 part Bi, 0.5 Pb, 0.25 Sn, 0.25 Cd), and finds the lead from the increased weight of the regulus.

XXV.

Assay of Iron Ores by Heat.

ONE part of the ore, about a hundred grains, thoroughly pulverized, is mixed with from thirty to a hundred grains of calcined borax. The quantity of the latter varies according to the purity of the mineral, and increases when it contains many foreign admixtures.

If the ore contains sulphur, it ought first to be roasted, as was the case with the assays of other metals already described.

A Hessian crucible is then prepared with coal, as already directed in the description of those vessels under the head of *Utensils and Implements*. Into the hollow in the centre, the mixture of ore and borax is poured, and on that some charcoal powder; after which the cavity is entirely covered

with a piece of charcoal. The crucible is then closed, the number of the assay being marked on the inner surface of the cover. This is then fastened down air-tight with some putty.

The iron ore, after these preliminary arrangements, is to be submitted to a reduction process in the furnace, fig. 6, Plate I., which lasts about three-quarters of an hour.

At the extensive iron-works on the Hartz Mountains in the interior of Germany, a very simple apparatus is used instead of the other furnace, of which, on account of its portability, I here give a description. It consists simply of an open cast-iron pot or jar, measuring about a foot across, and one and a half in height, and having a plate of sheet-iron, perforated with many little holes an inch in diameter, instead of a grate. This leaves a vacant space of about two inches below, to receive the ashes. The latter partition has two open

ings; one, the door to take out these cinders—the other, to introduce the end of the bellows. Above the iron plate, on which the crucibles are directly placed, without any brick, a coating of fire-clay, an inch thick, extends to the mouth of the jar.

The latter is furthermore supplied with three or four short legs, and a handle on each side.

In this furnace the process lasts about an hour and a quarter.

In both cases, the button produced contains exactly the same impurities, carbon, earths, acids, or other metals, as pig-iron would, if procured from the same ore, and therefore this assay is only to be used for furnaces. For these it entirely suffices; but to ascertain the true amount of pure iron, the wet analysis should be resorted to, the same as with other ores; and it is for this that I shall give directions in the next chapter how to discover or calculate the real per-centage of the pure metal, or to

assay the pig-iron produced in the process by heat.

It may not be uninteresting to many, who may honour these pages with their perusal, to become acquainted with a method for obtaining chemically pure iron, as given by Karsten in his famous and voluminous works on this metal. It is described in vol. i., pages 167, 168. He says, to procure chemically pure iron, take the best bar-iron of commerce, e. g. Swedish iron in the shape of small wire; cut it up in short pieces, and then mixing it with about a fourth part by weight of oxide of the same metal, melt it in a Hessian crucible. To cover it, use a compound flux, consisting of pure quartz, pure lime, and equally pure carbonate of potash, in proportions capable of furnishing a glass or slag, not flowing too easily, but of rather a tough consistency.

For the manufacture of an oxide of iron, free from all admixtures of foreign metals,

it is best to use small clean wire, oxidizing it by vapours of water.

The button thus produced has a remarkably white colour, a strong metallic lustre, and is more ductile than the best varieties of soft bar-iron. Particularly characteristic, however, is its great specific gravity, amounting to 7.9654, while that of wrought-iron is 7.6 to 7.9, and of cast-iron only from 7.0 to 7.5. The only impurity this metal may be discovered to possess occurs as slight traces of silicium, and therefore it may be considered to be as pure as it possibly can be made, even by a process conducted only upon the rules and principles of wet analysis, and thus in this shape it is utterly unknown to the mere practical metallurgist or smelter.

XXVI.

Fuchs's Process for Iron Ores and Iron.

THIS method, though remarkable for its simplicity, is a very sure one, not only to ascertain the quantity of iron in an ore, but to acquaint us with the peculiar degree of oxidation in which it exists, and also the amount of each oxide, which it is often not only very interesting, but useful to know. Many ores, particularly the magnetic ones, contain both the peroxide and the protoxide, and the only ones to which this process cannot be applied are those containing arsenious acid, not a very common ingredient.

The process discovered or invented by Fuchs is founded on the fact, that chemically pure muriatic acid, when atmospheric air is excluded, is incapable of dissolving any

copper; but that, when peroxide of iron is contained in it, a corresponding quantity of that metal becomes soluble, a muriate or chloride of iron and chloride of copper being thus formed.

The ore is dissolved in muriatic acid, and, if necessary, filtered. A small round-bottomed vial should be used. Into the solution a clean weighed strip of copper is placed, and the vessel corked, and covered with a piece of bladder, tied down round the neck until we are ready to boil it. This is best done in a water-bath, and should last until no more copper is received by the acid. The former is then taken out, well washed in water, dried with a towel, and immediately weighed.

The difference in the weight of the copper is all that is required to ascertain the amount of peroxide of iron in the ore, for we need only multiply this by the equivalent of peroxide of iron, which Fuchs takes to be 40, and divide the product by

31.7, the equivalent of copper. The quotient gives the quantity of peroxide contained in the solution or ore.

To know the whole amount of iron in it, we need only weigh off another part, (10—15 grs.,) dissolve it in muriatic acid, and then digest with chlorate of potash, to transform the protoxide into peroxide, after which, copper will decompose the whole. Instead of 40, the equivalent of peroxide of iron, we insert 28, the equivalent of the pure metal, into our calculations. By subtracting the amount of the peroxide from the last result, the amount of the protoxide is found.

The presence of alumina, silica, &c. has no effect on this assay, though the existence of copper in it would represent the ore as poorer than it really is. The latter, however, rarely occurs in common iron or its ores except in mere traces, and, as such, it produces no material difference.

XXVII.

Swedish Assay of Iron Ores.

THE following dry method of assaying iron ores is still much employed in Swedish works, and though the results obtained are less accurate than most of those obtained in the wet way, they approximate much more nearly to the economic value of an iron ore when smelted on a large scale. The yield of crude iron from the ores, as well as the approximate content of iron, is obtained by multiplying the weight of the resulting button of crude iron by 0.95. Only iron ores very rich in manganese have to be directly assayed as to their content of iron. The ores delivered at the works are first assayed in the dry way in order to determine the yield of iron, the nature of the iron, the behavior in smelting, etc.

The required assay-sample is obtained by taking pieces up to 200 lbs. or more in weight from the circumference and interior of the ore-pile and reducing them to powder. The powder, thoroughly mixed, is formed into a conical heap, which is divided into several parts. One or two of these parts are then taken and again formed into a heap, which is divided as before. This reduction is several times repeated until finally 2 to 4 lbs. of the sample remain, which are passed through a sieve having 40 to 60 meshes to the linear inch, and dried at from 230° to 248° F.

The aim in executing the assay is to reduce the oxidized iron, to carbonize the reduced iron, and to melt to a slag the admixed earthy matter. To effect the latter, silica, alumina and lime must be brought together in suitable proportion about 56 : 14 : 30 (bisilicate). In order to approximate this proportion, it is best to make a preliminary assay in the wet way for the de-

termination of the earthy matter present. For this purpose Berthier recommends the following process:—

A weighed quantity of the finely powdered ore is heated to redness in a porcelain crucible; the loss of weight gives the amount of water, carbonic acid, and other volatile matters present. Another weighed portion is heated with dilute nitric acid, which dissolves out carbonates of lime and magnesium; the residue contains, under ordinary circumstances, only oxide of iron, clay and quartz, the difference giving the amount of the earthy carbonates. A third portion is treated with strong hydrochloric acid, whereby the carbonates of lime and magnesium and the oxides of iron ore are dissolved out, leaving an insoluble residue of quartz and clay. This is weighed, the oxides of iron being determined from the difference of weight by deducting that of the carbonates previously found. From these results the proportion of fluxes neces-

sary to be added can be approximately determined, with a view to produce an easily fusible slag.

In many instances, however, this preliminary assay can be dispensed with, as a sufficiently good idea of the nature of the fluxes to be added can be determined by the appearance of the ore alone. Calcareous ores effervesce with acids; clayey ores, when breathed upon, give the characteristic odor of clay and adhere to the tongue, while siliceous ores give sparks when struck with the steel. According to experience, the following quantities of flux suffice in many cases :

| MATERIALS. | Chalk, per cent. | Fluor- spar, per cent. | China clay, per cent. |
|------------------------------|---------------------|------------------------------|-----------------------------|
| For magnetic iron ore..... | 10 | 25 | |
| “ specular iron ore..... | 5 | 25 | |
| “ red hematite iron ore..... | 25 | 25 | |
| “ brown iron ore..... | 20 | 20 to 25 | |
| “ argillaceous iron ore..... | 25 to 30 | 25 to 30 | |
| “ bog iron ore..... | 25 to 30 | 25 to 30 | |
| “ spathic iron ore..... | | 20 to 25 | 10 to 15 |
| “ sphaerosiderites..... | 20 to 25 | 25 | |
| “ finery cinders..... | 20 to 25 | 20 to 25 | |

According to the above table, for siliceous and argillaceous iron ores a flux of 20 to 25 per cent. of chalk and 20 to 30 per cent. of fluor-spar is as a rule sufficient. However, in many cases, especially with poor ores, the flux will have to be increased to 50 per cent. or more. When alumina is wanting, 5 to 15 per cent. of China clay is added. From the character of the slag obtained in melting it can readily be recognized whether the correct proportion of earths has been used. If the slag is translucent, has a gray, yellowish or violet (not green) color, and is enamel-like with conchoidal fracture, lime, clay and silica are in the proper proportions. If there is too much silica in the flux, the slag has a greenish tint, is glassy, more or less completely transparent, easily broken with a conchoidal fracture and sharp edges. Should the charge be fritted, pulverulent, and the iron diffused in minute particles, the flux has been wrongly proportioned, or the temperature too low.

This is apt to happen with ores containing a large proportion of lime and magnesium, as they are always refractory, and require the addition of a large proportion of silica and lime. If the slag is but half fused and dark in color, the ore contains an excess of silica or silicates of iron and manganese, which react on the carburetted iron, decomposing it, forming malleable iron and carbonic acid, giving it a spongy appearance. In this case lime should be added. The presence of a small amount of manganese is indicated by the amethystine color of the slag; larger amounts render it olive green, yellowish or brown. Titaniferous ores are apt to render the slag copper-colored by the formation of cyano-nitride of titanium. Chromic iron ores produce a resinous and dark-colored slag which is sometimes surrounded with a thin metallic layer. A stony, earthy slag with rough fracture and a gray, yellow or brown color, indicates a large content of bases. With a considerable

excess of lime the slag on being touched crumbles to a powder. Such basic slags with a stony texture may correctly give the content of iron if the button is not too graphitic. Iron ores containing sulphur may also be intentionally charged so as to obtain a basic slag in order to learn whether the iron button proves to be sufficiently free from sulphur. Thus Percy, for instance, recommends the formation of a slag with 38 Si O₂, 15 Al₂ O₃ and 47 Ca O, or nearly 2½ parts Si O₂, 1 part Al₂ O₃ and 3 parts Ca O. Such slag is almost formed by melting together the following fluxes :

| | |
|-----------|--|
| Glass 2½ | } Si O ₂ 1.75 { 35 other constituents = Al ₂ O ₃ 0.75 { 15 |
| Lime..... | |
| | 2.50 { 50 |

The following fluxes have also been recommended as adapted to ores or metallurgical products, which would be classed under the following several general heads. The proportions given are for 0.65 gramme (10 grains) of ore :—

1. Ores nearly free from gangue, some varieties of magnetite, red and brown hematite, specular iron ore and micaceous iron ore :—

| | Parts. | | Parts. |
|-----------------|---------|---------------------|---------|
| China clay..... | 2 | Lime..... | 2½ to 3 |
| Lime..... | 2½ | Blast-furnace slag. | 5 |
| Sand..... | 0 to 1 | Fluor-spar..... | 5 |
| Glass..... | 2 to 2½ | | |

2. Ores, etc., containing silica, varieties of limonite or brown iron ore, refinery slag (tap), and flue cinders :—

| | Parts. | | Parts. |
|------------|---------|-----------------|---------|
| Glass..... | 1 to 2½ | China clay..... | 2 |
| Lime..... | 2¼ to 4 | Lime..... | 3½ to 4 |

3. Ores containing carbonates of lime, magnesia, protoxide of manganese, etc., calcareous hematites, and spathic ores :—

| | Parts. | | Parts. |
|-----------------|--------|------------|---------|
| China clay..... | 2 | Glass..... | 3 to 4 |
| Lime..... | 1½ | Lime..... | 1½ to 2 |
| Sand..... | 1 | | |

4. Ores containing silica and alumina, clay iron ores, etc. :—

| | Parts. | | Parts. |
|------------|---------|-----------------|--------|
| Glass..... | 0 to 2½ | China clay..... | 0 to 2 |
| Lime..... | 2½ to 3 | Lime..... | 2 to 3 |

5. Titaniferous ores, or ores containing titanium :—

| | Parts. | | Parts. |
|-----------------|--------|------------------|--------|
| Lime..... | 3 | Glass..... | 2½ |
| Glass..... | 2 | Lime..... | 1½ |
| China clay..... | 2 | China clay | 1 |
| Lime..... | 2½ | | |

The dry assay of iron ores is performed as follows: Mix the finely pulverized ore with the required quantity of flux and transfer it to a basqued or charcoal-lined crucible. The basqued crucibles are prepared as follows: The charcoal powder is mixed with just sufficient gum-water or molasses to make it cohere readily. The crucible is gently rammed full of this charcoal, and a cylindrical cavity of sufficient size to contain

the charge is made in it with a spatula or some other boring instrument. The crucibles best adapted for this assay are made of a mixture composed of two parts unburnt and one part burnt clay. They are about $1\frac{1}{4}$ inches high and $1\frac{1}{4}$ inches in diameter at the top. As the amount of ore in the charges never exceeds from 0.64 to 0.96 gramme, four crucibles are placed in the furnace at a time. The charges in each are exactly alike, and if there is but a slight variation in the weight of the resulting buttons, the assay is probably correct; the mean weight of the four should be taken as the result. It is generally best to stand the four crucibles on a half brick, or lute them to it, so that when the fusion is finished all the crucibles can be removed from the furnace at one time. Although lids are sometimes used to cover the crucibles, it is better, after the charge has been introduced, to stop the cavity with a charcoal plug and to cover the entire top of the crucible with a clay

luting. The half brick holding the crucibles should be placed near or directly upon the fire-bars at the bottom of the furnace and the anthracite or coke fire built up around and over the crucibles. When the latter have been in the fire about $1\frac{1}{2}$ to 2 hours, and a white heat has been reached and maintained through a considerable part of this period, the assay may be considered finished and the crucibles removed. When the crucibles are cold they are broken up, the metallic buttons and globules * which may adhere to the charcoal lining and slag are separated out with a magnet. The appearance of the slag, which has previously been referred to, as well as the character of the iron buttons, serve for judging the assay. If the ores are easily reducible and the proper temperature has been reached and maintained in the furnace, the buttons

* The formation of these globules is prevented by not allowing any fine particles of charcoal or dirt to get into the cavity when it is being charged.

should be dark, tough and, if they have not been cooled too quickly, graphitic. As a general thing the iron buttons are dark gray or mottled according to the condition and amount of carbon they contain. If the crucibles are allowed to cool very slowly, the buttons are, as a rule, very graphitic, but if cooled quickly are fine-grained and white, or grayish in color. The presence of phosphorus generally renders the buttons white, hard and brittle; sulphur produces a mottled or reticulated structure; manganese, a hard, white crystalline or close-grained iron; titanium, a dull, dark-gray button with a crystalline fracture; chromium, a well-fused button, with a tin-white, bright crystalline fracture, to a semi-fused, white, or light gray, spongy mass, according to the amount of chromium in the iron.

A content of sulphur may also be recognized by the odor of sulphuretted hydrogen, or the brownish coloration of a lead-paper on

pouring hydrochloric acid over the regulus. By bringing a sheet of silver over a weighed quantity of the button thus treated, the former is colored the darker the more sulphur is present. By the degree of coloration it may, with some experience, be judged whether the iron is suitable for this or the other purpose.

In England the assay of iron is also executed in large unlined crucibles by mixing the ore with the previously mentioned fluxes (glass and lime) and pulverized charcoal, coke or anthracite. The ore and fluxes should be pulverized and passed through a 60-mesh sieve. With a series of four, the ore and fluxes may be proportioned as follows:—

1 and 2.

| | | |
|-------------|------------|----------------|
| Ore | 500 grains | (32 grammes) |
| Glass | 250 " | (16 grammes) |
| Lime | 300 " | (19.2 grammes) |

3 and 4.

| | | |
|------------------|------------|----------------|
| Ore | 500 grains | (32 grammes) |
| China clay | 200 " | (12.8 grammes) |

| | |
|-----------|--------------------------|
| Sand..... | 50 grains (3.2 grammes) |
| Lime..... | 250 " (16 grammes) |

The *reducing* agent employed is preferably anthracite, but charcoal or coke may be used. In any case it should be pulverized and passed through a 60 mesh sieve. The following amounts are respectively used :

- | | |
|-----------------------------|------------------------------|
| 1. 80 grains (5.12 grammes) | 3. 120 grains (7.68 grammes) |
| 2. 110 " (7.04 grammes) | 4. 150 " (9.60 grammes) |

Mix the fluxes and reducing agents thoroughly with the ore, transfer to a crucible about 4 inches high by $1\frac{1}{2}$ inches in diameter. Lute the cover on with clay, place on a piece of fire-brick and heat in the usual manner for an hour or more. In lifting the crucible out of the furnace, care must be taken to shake it as little as possible in order to avoid disseminating globules of iron in the slag. When cold, break open the crucible, and collect any small buttons or shots of metal in the slag with a

magnet. Weigh, then fracture the largest button and note the character of the iron. If the above scheme has been carefully carried out, using an ordinary hematite or magnetic ore, the results will be something like as follows :—

1. Not being sufficient carbon present, the ore is not all reduced ; the reduced and unreduced ore being fritted together with slag in an irregular lump.

2. Ore completely reduced and fused into a button. *Iron*, gray. *Slag*, glassy, and transparent, or perhaps dark-grayish, and opaque.

3. Ore completely reduced and fused into a well-melted button. *Iron*, gray to grayish white. *Slag*, clear, glassy and transparent ; color, gray to greenish by transmitted light.

4. Ore completely reduced, but owing to the excess of carbon present the metal is disseminated through the slag in small

shots or globules. *Iron*, gray. *Slag*, glassy, opaque or translucent.

There should always be present an excess of the amount of carbon necessary to reduce the ore. 100 parts by weight of sesquioxide of iron require $22\frac{1}{2}$ parts of carbon for reduction. Consequently the amount added must be adjusted according to the oxide of iron present.

XXVIII.

Assay of Quicksilver Ores.

THIS metal is one of those which sometimes occur in the virgin state, though by far the most common shape in which it presents itself is the bisulphuret, commonly termed cinnabar, which in its perfectly pure state consists of:—

| | | |
|---------|---------|---------|
| Mercury | | 86.287 |
| Sulphur | | 13.713 |
| | | <hr/> |
| | | 100.000 |

Its formula being Hg S. A specimen from Japan, analyzed by Klaproth, gave:—

| | | |
|---------|---------|-------|
| Mercury | | 84.50 |
| Sulphur | | 14.75 |
| | | <hr/> |
| | | 99.25 |

Which, though it must have contained

0.75 of other ingredients not mentioned in the analysis, shows that it possessed as much as, according to the above-calculated composition, it could be expected to contain.

It is not the place here to give the analyses of other casual occurrences of this metal, such as perhaps are only to be found in the cabinet of the mineralogist; and I shall therefore proceed to describe the manner in which the assays of mercurial ores are performed.

In all cases the mercury is procured by simple distillation, for which purpose, when not originally uncombined, it is necessary first to disengage it.

In distilling quicksilver, an iron retort covered outside with a coating of clay is commonly used. The neck should be so contrived that it can be unscrewed, to facilitate cleaning the interior. Retorts or tubes of clay, glazed externally, are also applicable, and are preferred by some; but

those of iron are safer, inasmuch as the vapors of mercury cannot possibly penetrate and escape through them.

To produce the necessary heat, which should not exceed a mild red, any convenient furnace, such as the one described on page 220, will answer.

Any tumbler or beaker-glass containing cold water may be employed as a condenser. It must, however, be remarked here that the mouth of the retort ought not to be inserted into the water, because in that case, on cooling, the water would rise up into it. To avoid this, and yet to prevent the escape of the mercurial vapors, a wet wrapper of paper or linen is bound round the neck of the retort, protruding sufficiently to permit of the other end being completely immersed in the water. Some assayers employ a little bag tightly attached to the retort. .

The fluxes used are various, and, according to their efficiency, may be enumerated thus, the first being the best:—

Black flux, from one-half to full weight of ore
Carbonate of soda or of potash, one-half “ “
Iron filings, from a quarter to a half “ “
Carbonate of lime, one-half to full “ “
With charcoal powder, one-tenth to one-eighth “

The black flux produces with cinnabar besides liberating the mercury, sulphuret of potassium, while sulphates of soda and of potash are formed from the carbonates. The iron filings simply cause the formation of protosulphuret of iron, while the quicksilver is disengaged. The alkaline fluxes are chiefly used with the iron retorts. If the quicksilver is known to exist only in its virgin uncombined state, no flux is required, and we may immediately proceed to distil it over.

It has been remarked above that the heat employed during the assay should not exceed a moderate red. This point is of great consequence with cinnabar, because this sulphuret is capable of being sublimated in its undecomposed state, if the heat is increased beyond that temperature. If the

ore contain chloride or bromide of mercury, these, too, will pass over; and, to avoid this, the ore, if their presence is suspected, must first be mixed with soda. To render the mixing more intimate, a little water is employed, which must, however, be removed before placing the ore in the retort. It is also advisable to apply the heat gradually, and, when retorts are used, to be particular that they are everywhere heated equally, as otherwise quicksilver might be condensed in the interior of the retort. When it has been ascertained that drops of quicksilver are no longer leaving the retort or tube, and therefore that the process is about being completed, it is advisable to fire up a little before removing the receiving vessel, so that particles of the metal condensed in the neck of the retort or in the tube may be forced out, though, to insure this still more, it is necessary to brush out the neck of the vessel with a little brush or feather.

The quicksilver which has collected in

the condensing vessel is frequently indisposed to join and form a connected mass; but this is easily brought about by boiling it in water. Adhering moisture remaining when the water is decanted should be removed with blotting-paper.

From one-half to three pounds of the ore are commonly employed for the assay, decreasing in quantity as the value of the ore increases.

Perfect accuracy can never be attained in the assay of quicksilver ores by heat, neither as regards the perfect chemical purity nor the full amount of the metal produced; but, as the loss and impurity are yet greater with quicksilver produced on a large scale, this method of treating the ore will be sufficient for technical purposes.

Berthier, in the *Annales des Mines*, iv série, t. iii. p. 820, suggests that when the ore is a very poor one, and when therefore the large amount it would be necessary to use might be the cause of considerable in-

convenience in the assay, the following preparatory process be resorted to. The ore should be digested with aqua regia, the supernatant liquid decanted or filtered off, the residue thoroughly washed with water, and the acid solution, together with the washings, evaporated to dryness. All the quicksilver in the shape of chloride is then contained in the last-dried residue, and can be further treated in the dry way, only that, as above remarked, it is always necessary carefully to mix with soda, when choride of quicksilver is present.

Before closing this chapter it will be well to describe the treatment necessary when a quicksilver ore contains arsenic. I shall literally translate the remarks of Bodeman in the work quoted in the preface. He says:—

“An ore from Huanca-Vélica, in Peru, containing arsenic (red sulphuret of arsenic, &c.) and cinnabar, having been submitted to Berthier's inspection, he, after various

useless experiments, discovered the following process to be the most adequate for determining the mercury in it.

“The ore is mixed with four or five times its weight of litharge, and then heated in a retort. A flowing, slag-like mass is formed by the litharge, sulphuret of arsenic, &c., while the cinnabar is decomposed into sulphurous acid and metallic mercury. The quicksilver is completely volatilized by a moderate heat, and collected in the condensing apparatus and the farther portion of the neck of the retort. The only precaution necessary during the operation consists in gradually and but moderately heating the clay or glass retort, to prevent its being perforated by the effects of the litharge before the process is concluded.”

Electrolytic determination of quicksilver.
According to Escosura, 0.5 gramme of the ore is digested with 10 to 15 cubic centimeters of hydrochloric acid and 20 cubic centimeters of water in a porcelain dish.

After boiling add from 0.5 to 1 gramme of potassium chlorate in small portions. When decomposition is complete, dilute with 50 cubic centimeters of water and expel free chlorine by continued boiling. In order to separate selenium or tellurium, if present, add 20 to 30 cubic centimeters of a saturated solution of ammonium sulphate and boil moderately. When the insoluble residue has settled, filter, and use the filtrate as the electrolytic bath. The negative electrode should be pure gold, and the positive a platinum sheet. The solution is subjected to the galvanic current from 24 to 30 hours. The increased weight of the gold equals the content of quicksilver. Two Bunsen cells are generally sufficient. This assay may also be executed by treating the finely pulverized ore in a platinum dish with hydrochloric acid, ammonium sulphate, and water. Of 10 per cent. ore only 0.2 gramme is used, and of 0.1 per cent. ore, 10 grammes. The platinum dish

serves as the negative pole, and a disk of sheet-gold, about 4 centimeters in diameter, as the positive pole. The current is supplied by 6 Meidinger cells, and the quick-silver is precipitated in 24 hours.

These determinations are said to be very accurate and are exclusively used at Almaden.

From a solution of its salt acidified with nitric acid, mercury is, according to Classen, precipitated on the negative electrode in the form of a mirror, or of small globules, by a current of 0.2 to 0.5 cubic centimeter of oxyhydrogen gas. The metal adheres well and can be washed without loss. The washing must, however, be done without interrupting the current. Insoluble quick-silver compounds may easily be analyzed by suspending them in acidulated water, or in a dilute solution of common salt (1:10) and electrolyzing as usual.

XXIX.

Dry Assay of Tin Ores.

TIN that is to be assayed in the dry way must be present in the form of oxide. The assay proper consists of applying in a crucible at a high temperature reducing and fluxing re-agents which bring the stannic oxide to the metallic state and convert the gangue into a liquid slag. The behavior of stannic oxide and of metallic tin at an elevated temperature causes, however, certain difficulties, which make the results often less accurate than could be desired. These difficulties are :

1. That some of the resulting metallic tin is liable to be volatilized.
2. That metallic tin decomposes the alkali carbonates used as fluxes forming stannates which enter the slag.

3. That at the temperature required to reduce the stannic oxide, other metallic oxides become also reduced to the metallic state and alloy with the resulting tin button, and vitiate the assay.

4. That sulphates are reduced, or, if sulphides are present, they combine directly with the metallic tin and carry it into the slag.

5. That the gangue in the ore is always siliceous. This causes loss as the stannic oxide, while passing through the intermediate state of stannous oxide, combines with silica and silicates.

6. That stannic oxide is liable, before its reduction has begun, to combine with the basic fluxes used in assaying and form stannates; this liability increases as the quantity of flux is increased.

Thus it will be seen that the results can be too high (as when iron enters the tin button) or too low (as when tin is carried off in the slag as stannate, sulphide, or sili-

cate), and that all impurities should be removed, if possible, before the actual dry assay takes place. The making up of the charge must also be regulated to counteract as much as possible the bad influences of the basic fluxes. The crucibles used in assaying tin ores are the ordinary clay crucibles, the plumbago crucible, and the porcelain crucible. Crucible linings are unnecessary.

German method of assay. Five grammes of ore are intimately mixed with 0.75 to 1 gramme of charcoal dust, and charged into a clay crucible; on top are placed 12.5 to 15 grammes black flux (or the substitute: 2 parts of potassium-sodium-carbonate and 1 part of flour), with 1 to 1.25 grammes borax-glass, then a common salt cover, and finally a piece of charcoal. The crucible being covered, heat in a muffle or a pot-furnace at a moderate, gradually increasing temperature, until the boiling has ceased, and then for from half to three-quarters of an

hour at a white heat. The crucible is then removed from the fire, broken, when cool, and the tin button weighed.

Mitchell * mixes 400 grains ore, 100 argol, 300 sodium carbonate, 50 lime, and charges in a crucible so large that it need only be half filled; then gives a cover of sodium carbonate and 200 grains of borax, heats gently and keeps for at least 20 minutes at a dull red heat, increasing the temperature, until tranquil fusion takes place.

Leval's assay with potassium cyanide. A sufficient quantity of powdered potassium cyanide is rammed into a capacious porcelain or fire-clay crucible to form a layer of from 12 to 15 millimeters thick. Five grammes of the powdered ore intimately mixed with five times the quantity of potassium cyanide are added to that in the crucible, and the whole is covered with a thin layer of the cyanide. The charge is then

* Manual of Assaying; New York, 1881, p. 48.

heated in a moderate fire until it fuses, and is kept in constant fusion for 10 minutes. The crucible is then taken out and gently tapped to facilitate the formation of a single button, and allowed to cool. The button is then freed from adhering slag by water. In case copper or lead is present, the ore must be freed from them before reduction by treating it with acid. This is the most accurate method of assaying tin (to within one-half per cent.), and can be executed in a very short time. In case the ore is siliceous, a mixture of 10 grammes of ore, 3 to 8 grammes of ferric oxide, and 40 grammes of potassium cyanide, is placed in a crucible lined with charcoal. The mixture is first covered with potassium cyanide and then with powdered charcoal. The cover is luted on and the charge heated at a high temperature for one-half to one hour.

In any case it is always better to mix a small quantity of powdered charcoal with

the charge. One-half of the potassium cyanide may be mixed with the ore and the balance used to cover the charge. Care must be taken not to allow the fire to become too hot or the charge to boil over. After pouring, the mould must not be moved until the slag has set, otherwise it is apt to penetrate into the button.

Winkler's bronze assay. Five grammes of ore are mixed with 5 grammes of cupric oxide and charged in a clay crucible; on top are placed 15 grammes of black flux with 1.25 grammes of borax-glass, then a common salt cover, and finally a large piece of charcoal. The crucible is heated in a muffle or pot-furnace, after boiling has ceased, for three-quarters of an hour to an hour at a bright-red heat, finishing the assay at almost a white heat. The result is a white, brittle, tin-copper alloy. With every tin-assay, an assay of 5 grammes of cupric oxide with the same charge is made, and the weight of the resulting button of

copper, subtracted from the bronze-button, gives the weight of the tin contained in the ore.

This assay is actually a modification of the German method previously described. Its advantage is that no prills of tin (or, rather, particles of bronze) are found suspended in the slag. All the metallic oxides that have been reduced are found collected in one button. It is claimed that more tin is recovered by this method than by the regular German method, and that the buttons do not differ more than from 0.4 to 0.5 per cent.

XXX.

Assay of Sulphurets in Ores.

It is often very necessary to know the quantity of sulphurets contained in ores, to be able to arrange accordingly the processes of smelting them.

To one part of ore, 50 to 100 grains, one part of powdered glass and two of borax are required.

The borax is first mixed with the ore, and when placed in the Hessian crucible, or in the lead and copper one, fig. 4, Plate II., both are covered with the glass. The crucible is then exposed to a bright red-heat, as with copper and lead, and on cooling the button is broken out, which may contain in the shape of sulphurets, copper, lead, iron,

&c., according as these exist in the ore, and it may then be examined for these metals by the means already given in their respective assays.

Volumetric determination of sulphur in ores which contain either sulphur alone, or also sulphates. Weil gives the following method: Place 1 to 2 grammes of the finely powdered ore in a flask provided with a cork, through which passes a bent tube. The outside end of this tube dips into an ammoniacal solution of copper of known strength. A few small pieces of granulated zinc are placed in the flask, and 75 cubic centimeters of hydrochloric acid poured over its contents; quickly close the flask and heat. The sulphuretted hydrogen developed precipitates an equivalent portion of copper, and, when precipitation ceases, the sulphide is allowed to settle, is filtered and washed. The object of adding the zinc is to dilute the sulphuretted hydrogen with hydrogen, and to carry off the last

traces of it which may remain in the flask and glass tube. The amount of filtrate from the precipitated copper sulphide is carefully measured, and 10 to 20 cubic centimeters of it saturated with from 25 to 50 cubic centimeters of hydrochloric acid. Heat to boiling, and when boiling titrate with a standardized solution of stannous chloride. This quantity multiplied by 0.50393 gives the content of sulphur sought. The zinc added facilitates the action of the acid upon the ore, and if any galena is present, it is also more readily decomposed; the chloride of lead formed being reduced to metallic lead by the zinc.

Examination of sulphur in metallic lead.

Treat 20 to 30 grammes of very fine chips of the lead with a considerable excess of concentrated hydrochloric acid; sulphur will be set free as sulphuretted hydrogen. Pass this sulphuretted hydrogen, by means of an aspirator, into bromine water, in which it is decomposed, sulphuric acid be-

ing formed. Determine the sulphur in the latter with barium chloride as usual. The solution of the lead is aided by gentle heat. A large excess of hydrochloric acid prevents the separation of lead ehloride.

Determination of sulphur in pyrites. To 1 gramme of the finely powdered ore in a porcelain dish or casserole add a small amount of potassium chlorate, cover the dish with a watch glass, add 50 cubic centimeters concentrated nitric acid, and heat to boiling, adding a little chlorate from time to time. When perfectly oxidized, remove the watch glass (and it should be rinsed into the dish), and evaporate to a small bulk on a water bath. Add a little strong hydrochloric acid, and evaporate to dryness, moisten with the same acid, add water, and filter from silica and the gangue. To the filtrate add 1 gramme tartaric acid, heat, add hot solution of barium chloride, drop by drop, boil, let settle, filter and wash well with hot water.

Weigh a clean porcelain or platinum crucible, add the filter and precipitate, burn to ashes, cool, and weigh as baric sulphate. After deducting weights of crucible and filter ash, multiply the remainder by 0.1374, and the product by 100 for percentage of sulphur.

Bodewig's method of determining sulphur in pyrites. One-half gramme of pyrites is treated in a glass-stoppered vessel of about 100 cubic centimeters' capacity, with 30 cubic centimeters of water and 4 c. c. of bromine. The stopper is quickly inserted, and the vessel shaken for 5 minutes. When oxidation is ended, which is known by the disappearance of all pulverulent sulphur adhering to the sides of the glass, the solution is emptied into a casserole, and most of the bromine allowed to evaporate in the cold. The solution is almost neutralized with ammonia, then poured into an excess of hot ammonia contained in a platinum dish, and digested from 10 to 15 minutes at

a gentle heat. The sulphuric acid is determined in the filtrate from this in the usual manner. The entire amount of bromine employed must not be added all at once, as otherwise there may be some loss of sulphur in the form of sulphuretted hydrogen. Some iron volatilizes as a bromide with the excess of bromine; hence it cannot be determined in the precipitate made by the ammonia.

XXXI.

*Mode of testing the Calorific Power of Coal
and other Fuels.**

It must not be supposed that the amount of heat which a fuel is capable of producing is entirely dependent on its chemical composition.

Different means have been adopted to determine the efficacy of coal as a fuel. Thus, Despretz has made experiments of this kind by discovering the amount of water which a certain quantity of coal will elevate in temperature one degree of the centigrade thermometer. The varying temperature of the water, however, must ever prevent an accurate result. Rumford has proposed to determine the same by the

* Karsten's *Eisenhüttenkunde*, vol. ii., p. 219, § 476.

direct use of a thermometer in a closed vessel. Berthier has, however, proposed one that seems equally simple, and serviceable for all practical purposes.

According to his proposition, one gramme of the coal (or other fuel; for it is applicable to all) should be thoroughly pulverized. All coals are readily reduced to such a state. Wood should be used as shavings, or rasped. The powdered substance is then mixed with some litharge, but more than it is capable of reducing,—not less than twenty times its own weight nor more than forty. The quality of the fuel will enable the assayer, after a little practice, to determine *à priori* with considerable accuracy what will be the requisite quantity. The mixture is placed in a clay crucible (Plate II., fig. 4 or 5) and carefully covered with about twenty to thirty grammes of litharge. The crucible must not be filled over one-half. This is then placed in a heated muffle and covered. The heat should not be too

severe and rapid. The contents will boil up. When the melting has thoroughly taken place, strong heat should be applied for ten minutes, so that the button may collect. The crucible is then taken out and cooled slowly, then broken, and the button extracted and weighed.

This process rests upon the fact that the carbon will reduce the litharge; and having, therefore, assumed one number for the amount of litharge reduced, from which to calculate our experiments, it will be easy to compare the different varieties of fuel. When many experiments of this kind are made, the assayer will do well to establish a certain number as the basis. This will vary according to the general character of his coals. This number it will be well to assume as low as possible, to avoid the inconvenience of fractions. He would therefore adopt a very inferior variety of coal as the one with which to compare the others in the following manner, though

be it remembered I am not now giving results of assays, but only imaginary examples.

1 part bone-coal reduced 10.00 parts by weight of litharge

| | | | | |
|----------------------|-------|---|---|---|
| " rough bituminous " | 12.50 | " | " | " |
| " good bituminous " | 20.00 | " | " | " |
| " anthracite " | 25.00 | " | " | " |

Table of Analyses of different Clays, from which the most approved Crucibles are made.

| Locality, and name of analyzer. | Silica. | Alumina. | Magnesia. | Lime. | Perox. of Iron. | Water and Bitumen. | Carbon. | Total. | To what use applied. |
|--|---------|----------|-----------|-------|-----------------|--------------------|---------|--------|--|
| 1. From Stourbridge, England, by Berthier. | 63.7 | 20.7 | — | — | 4.0 | 10.3 | — | 98.7 | The two first clays, 1 to 3 inclusive, are used chiefly for crucibles in the manufacture of steel in England. |
| 2. Same place by Le Play. | 46.1 | 38.8 | — | — | — | 12.8 | 1.5 | 99.2 | |
| 3. From Stammerston, England, by Le Play. | 42.0 | 40.9 | 0.1 | 1.3 | Trace | 14.7 | — | 99.0 | |
| 4. From Almerode, Hessa, by Berthier. | 46.5 | 34.9 | — | — | 3.0 | 15.2 | — | 99.6 | This is the clay from which the famous Hessian crucibles are made. |
| 5. From Passau, by Leshen. | 42.4 | 57.6 | — | — | 0.7 | — | — | 100.7 | These two, the first a clay, the second, graphite, are mixed in the proportions of from 3 to 4 of the first to 1 part of the latter, in the manufacture of the well-known Passau graphite crucibles. |
| 6. Graphite from same place, by Berthier. | 41.2 | 14.7 | 1.0 | — | 8.2 | 1.0 | 33.9 | 100.0 | |

Table showing, in Degrees of the Centigrade and Fahrenheit Thermometers, the Amount of Heat necessary to melt various Substances.

| | | | | Centigrade. | Fahrenheit. |
|-------------------------------|--------------------------|-------------------------|--|-------------|-------------|
| Platina | | | | 2500 | 4532 |
| Bar Iron | | | | 1500-1600 | 2732-3012 |
| Steel | | | | 1300-1400 | 2372-2552 |
| Cast iron, gray..... | | | | 1200 | 2192 |
| Do. white..... | | | | 1050 | 1922 |
| Gold..... | | | | 1100-1250 | 2012-2282 |
| Silver..... | | | | 1000 | 1832 |
| Bronze..... | | | | 900 | 1652 |
| Antimony..... | | | | 450 | 842 |
| Zinc..... | | | | 360 | 680 |
| Lead..... | | | | 330 | 658 |
| Bismuth..... | | | | 260 | 480 |
| Tin..... | | | | 230 | 446 |
| Alloys of Tin, (in parts.) | Lead, and (in parts.) | Bismuth. (in parts.) | | | |
| 1 | 3 | - | | 289 | 554 |
| 1 | 1 | - | | 241 | 464 |
| 3 | - | 1 | | 200 | 392 |
| 3 | 1 | - | | 186 | 365 |
| 2 | - | 1 | | 167.7 | 333.5 |
| 1 | - | 1 | | 142.2 | 288.8 |
| 4 | 1 | 5 | | 188.9 | 246.2 |
| 3 | 2 | 5 | | 100 | 212 |
| 3 | 5 | 8 | | 100 | 212 |
| 1 | 1 | 4 | | 94 | 203 |
| Sulphur..... | | | | 109 | 230 |
| Mercury..... | | | | -39 | -38.2 |

*Table of Troy Weights used with Gold, and Silver, and
Platina.*

24 grains (gr.) make 1 pennyweight (dwt.)

20 pennyweights — 1 ounce (oz.)

12 ounces — 1 pound (lb.)

| lb. | oz. | dwt. | gr. |
|-----|------|-------|--------|
| 1 | = 12 | = 240 | = 5760 |
| | 1 | = 20 | = 480 |
| | | 1 | = 24 |

The value of gold is given in carats fine, 24 c. f. being pure.

One pound of gold 24 carats fine contains 5760 grs. of pure gold, as gold of that number of carats fine is unalloyed.

One pound of gold 23 carats fine contains 5520 grs., and so on, and one oz. of gold 20 c. f. has 400 grs.

one dwt. of gold 15 c. f. has 15 grs.

These are given as examples of the manner in which the amount of the pure metal ought to be calculated.

Table of Avoirdupois Weights used with other Metals.

| (Tun.) T. | (Hundredweight) cwt. | (Quarter) qr. | (Pound) lb. | (Ounce) oz. | (Dram) dr. |
|--------------|-------------------------|------------------|----------------|----------------|---------------|
| 1 | = 20 | = 80 | = 2240 | = 35840 | = 573440 |
| | 1 | = 4 | = 112 | = 1792 | = 28672 |
| | | 1 | = 28 | = 448 | = 7168 |
| | | | 1 | = 16 | = 256 |
| | | | | 1 | = 16 |

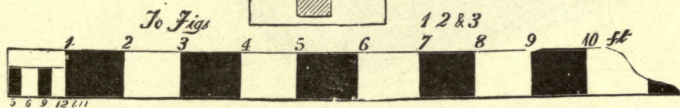
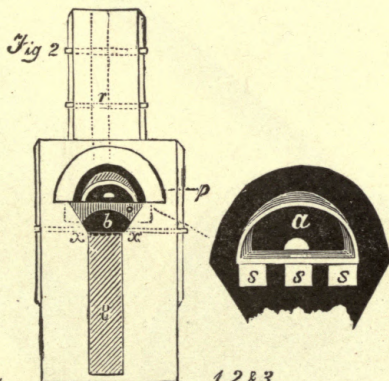
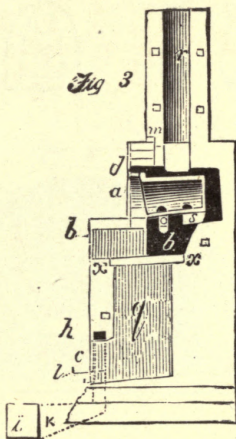
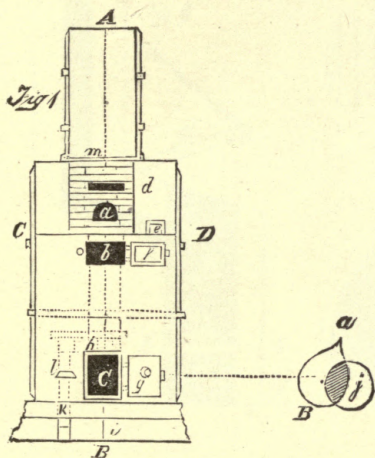
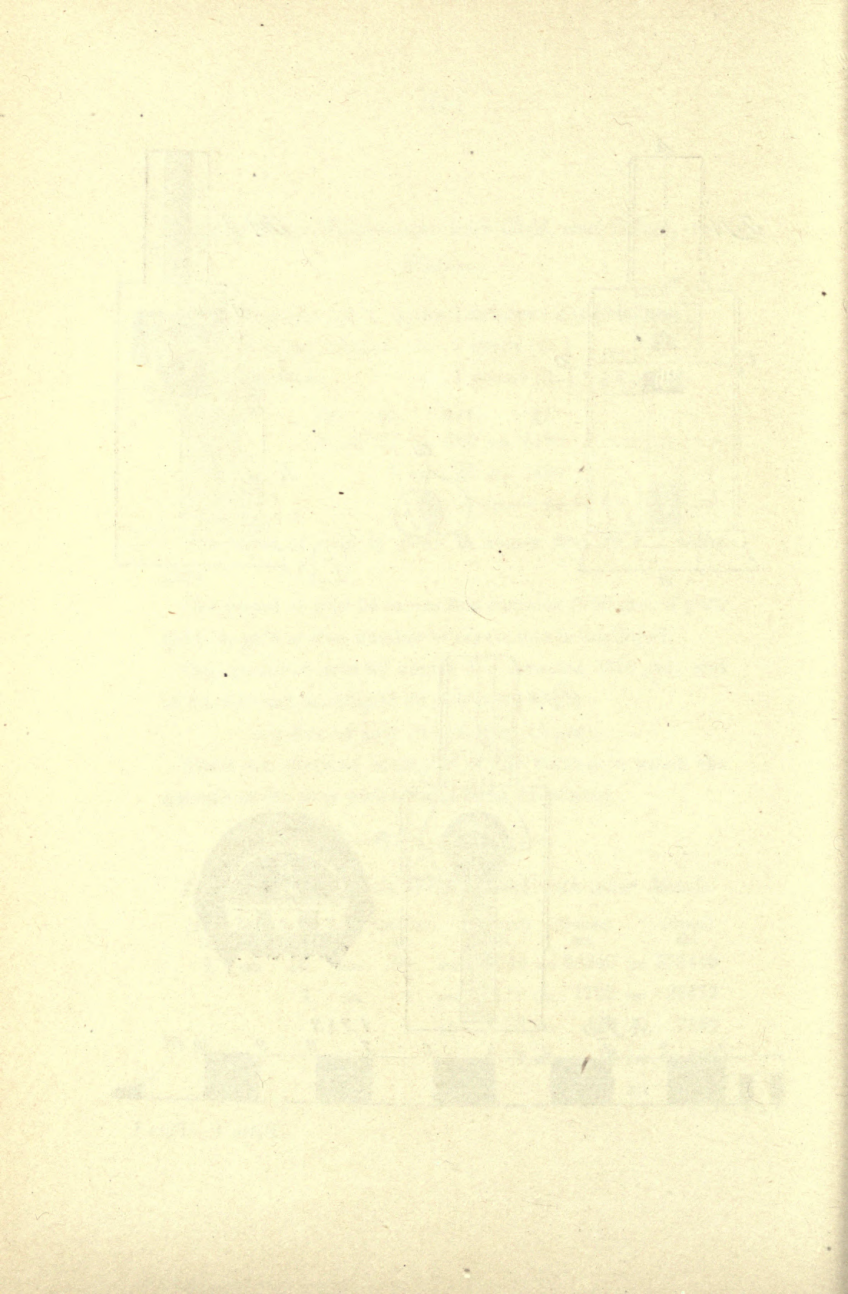


Plate I.—Part I.



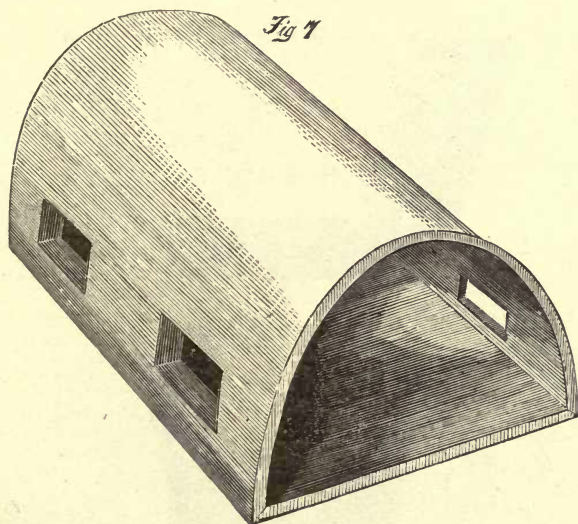
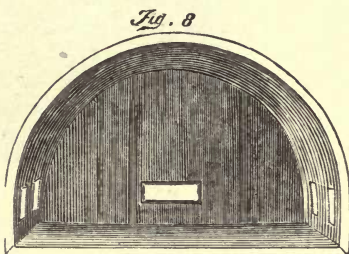
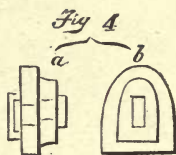
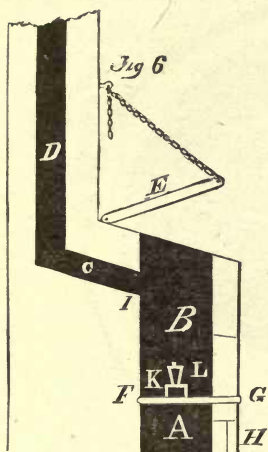
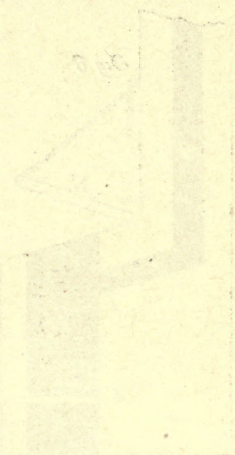


Plate I.—Part II.



1871-1872

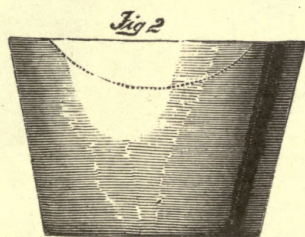
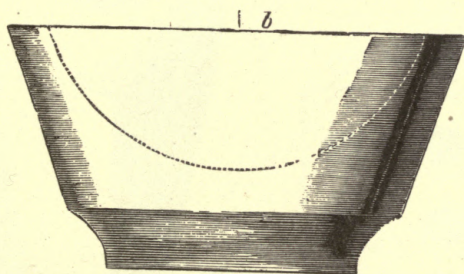
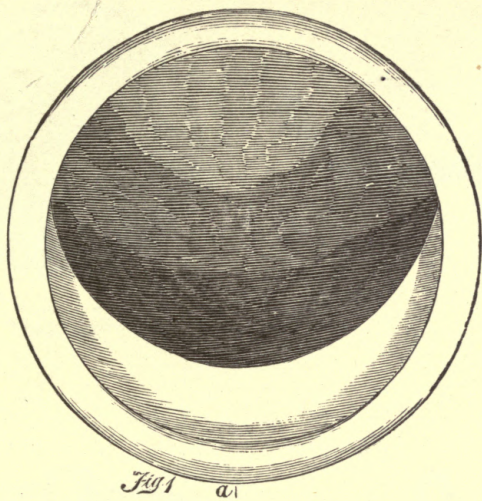
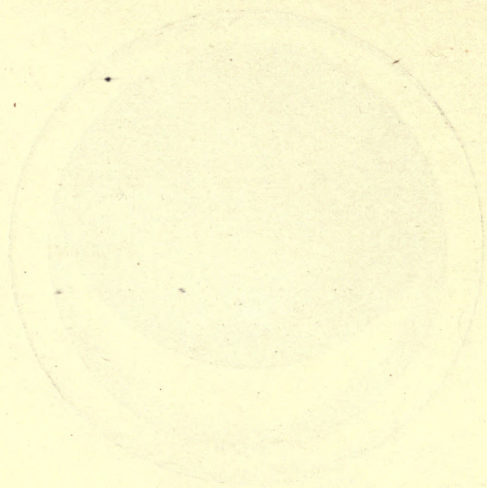


Plate II.—Part I.



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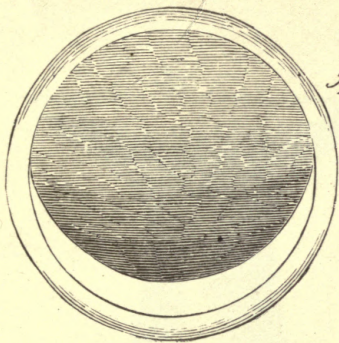
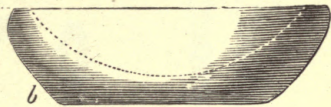


Fig 3



a

b

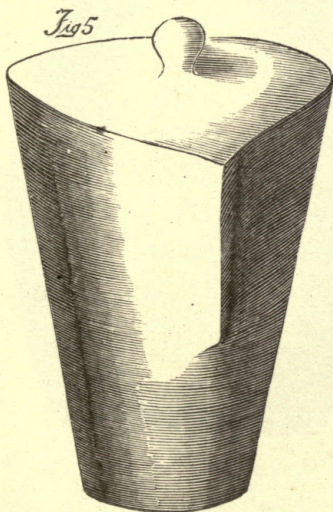


Fig 5

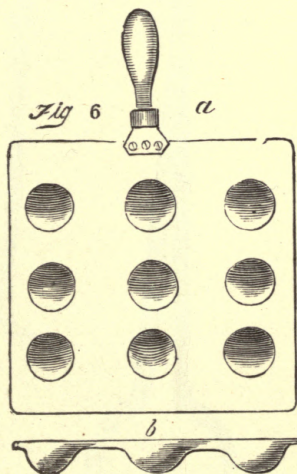


Fig 6

a

b

Plate II.—Part II.



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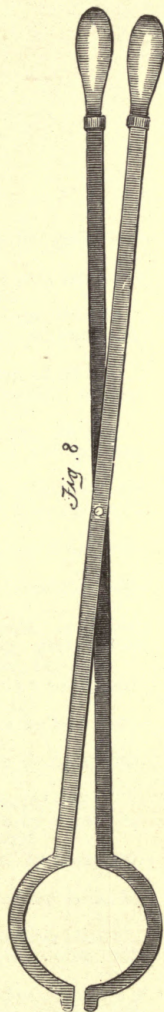
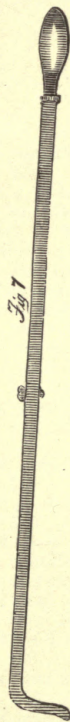
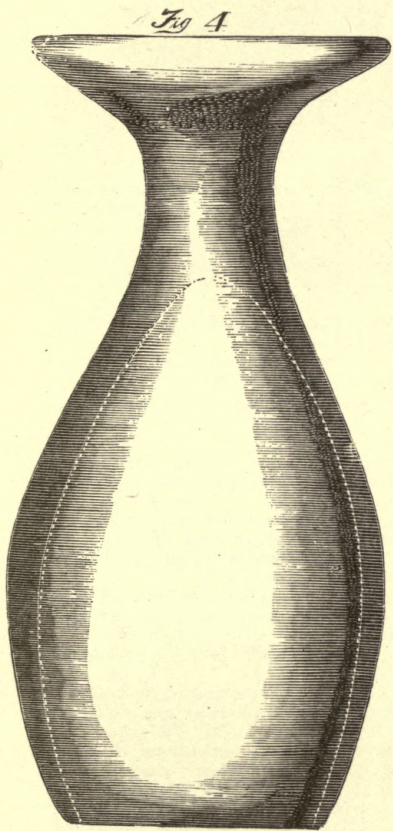


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